TEXTILES PAPER LEATHER

AMERICAN DYESTUFF REPORTER

Including the Proceedings of the
AMERICAN ASSOCIATION OF TEXTILE CHEMISTS AND COLORISTS

VOLUME XIV NOVEMBER 16, 1925 NUMBER 19

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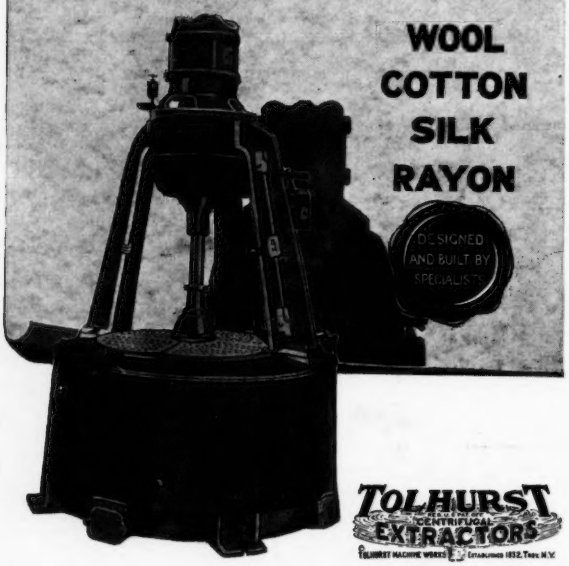

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4. Any weight of yarn up to 100 lbs. may be dyed on one machine.
5. The essential parts are in Liberty Silver, which is satisfactory for all dyestuffs
6. The fastest known colors, including the Naphthanil and Vat colors, can be satisfactorily dyed on this apparatus.
7. The dyeing is carried out in full view of the operator.
8. The machine is mechanically simple and automatic in its action.

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Total annual capacity of machines at work exceeds One Hundred and Fifty Million pounds.

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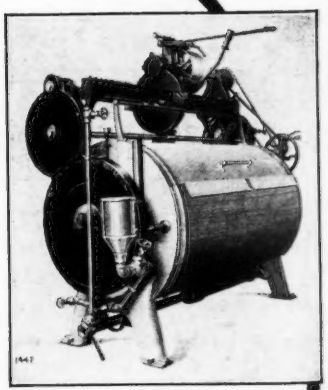

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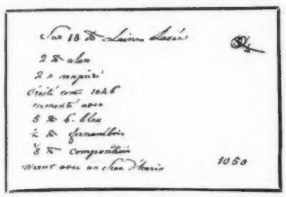
Recipe from the Ancient French Dye Recipe Book

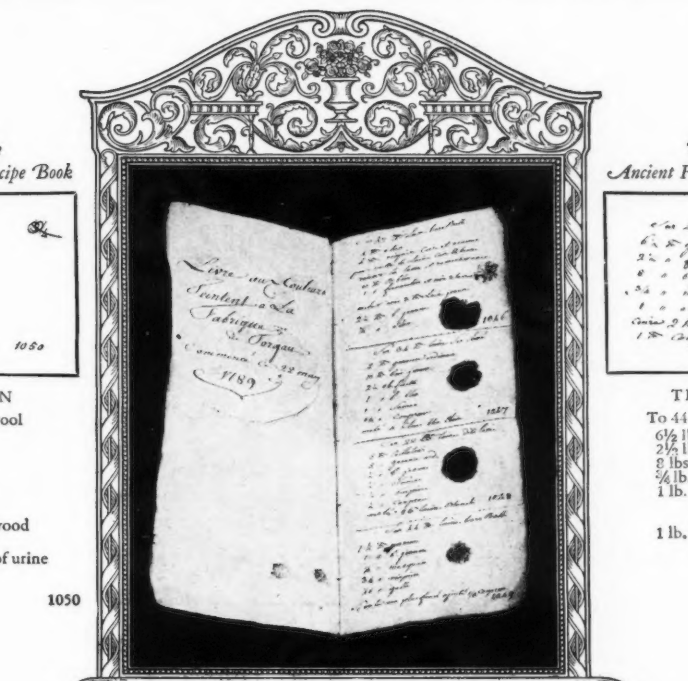


TRANSLATION

To 18 lbs. washed wool
2 lbs. alum
2 lbs. vinegar
treated as 1046

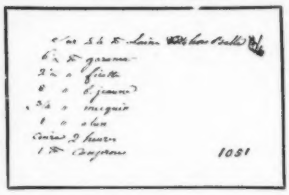
Topped with
5 lbs. Blue wood
½ lb. Pernambuco wood
⅛ lb. composition
soften with one pail of urine

1050



This ancient French dye recipe book is now in possession of the General Dyestuff Corporation. Among other recipes of historical interest it contains the formulae for the world famous French Gobelins.

Recipe from the Ancient French Dye Recipe Book



TRANSLATION

To 44 lbs. Wool out of bale
6½ lbs. crapp
2½ lbs. ficette
8 lbs. yellow wood
⅝ lb. "Muquin"
1 lb. alum

Boil 2 hours
1 lb. Copperas

1051

No. 1 of a series of advertisements based on the dye recipes that established the supremacy of French textiles in the XVIIIth Century

WASHINGTON was inaugurated President when this collection of dyeing recipes was used for the production of fast colors in France. Now, after 136 years, these hand-penned formulae possess a deep historical and scientific interest. They graphically display the tremendous advance made by the dyestuff industry since 1789. Users of dyestuffs now have at their disposal many colors of far better fastness and easier method of application. The highest requirements of industry are today met by the types offered by us, and include the products of

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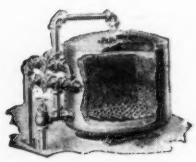

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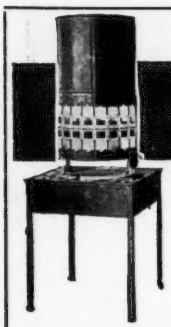

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Unevenly Dyed
"Three-Fibre Hose"
Streaked Dyeing and
Smears on the Finished Hose

are generally caused by the fact that oils used for winding Artificial Silk are

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"Circulated Everywhere Dyestuffs Are Used"

Devoted to the practical application of dyestuffs in all industries: especially to textile chemistry—the science and practice of scouring, bleaching, dyeing and finishing.

VOLUME XIV | NEW YORK, NOVEMBER 16, 1925 | NUMBER 19

Acetate Silk—Its Dyes and Their Application

SUPPLEMENTAL TO THE PRECEDING SERIES OF ARTICLES ON DYEING ACETATE SILK

Rayon Production—Preparation and Constitution of Cellulose Acetate—Catalysts—Identification—The Ruthenium Red Test—The Iodine-Zinc Chloride Test—The Acetate Test—Acetate Silk Saponified in Scouring—Regain and Strength—Mercerizing Celanese Unions—Bleaching Acetate Silk—Bluing or Tinting Acetate Silk White

By CHAS. E. MULLIN, M.Sc., F.A.I.C.

(All rights reserved by author)

NO other branch of the textile and dye industry is advancing so rapidly and attracting as much attention as that of acetate silk and its dyes. Since writing the preceding series of articles, "Dyeing Acetate Silk," so much additional information has come to hand that in order to bring the subject up to date and make it complete, a supplementary instalment is necessary.

Rayon Production

Possibly Reamer was the first to suggest artificial silks in 1734, but the first patent appears to be that of Audemars, a Swiss, who in 1855 attempted to utilize an ether-alcohol solution of nitrocellulose, prepared by nitrating the bast fibers of the mulberry tree. This effort was not successful, but in 1882 and 1883 Powell, Weston, Swan, Swinburne and Wyne worked out methods very similar to those later adopted by Chardonnet. Chardonnet had his first success in 1884 or 1885, but it was not until about 1890 that it was really successful commercially. Douglas Woolf in Textile World, 68, 1749-50 (1925), gives the following tables on rayon production:

TABLE NO. XIII

World Rayon Production by Years

(in pounds)

1914	26,000,000	1922	79,738,000
1918	35,000,000	1923	97,000,000
1919	40,000,000	1924	141,414,000
1920	50,000,000	1925*	185,000,000
1921	65,000,000		

*Estimated.

TABLE NO. XIV

World Rayon Production by Countries

(in pounds)

	1922	1923	1924
United States	23,500,000	35,400,000	39,000,000
England	15,340,000	16,500,000	23,947,000
Germany	12,584,000	13,000,000	23,672,000
Italy	6,292,000	10,000,000	18,480,000
France	6,292,000	7,700,000	12,333,200
Belgium	6,292,000	6,000,000	8,874,800
Switzerland	1,887,600	3,700,000	4,004,000
Holland	2,516,800	2,600,000	3,366,000
Austria	1,573,000	.....	2,640,000
Poland	943,800	.....	1,540,000
Czecho-Slovakia	629,200	.....	1,293,600
Japan	.....	.....	1,199,000
Hungary	1,887,600	.....	616,000
Spain	.....	.....	184,800
Sweden	.....	.....	176,000
Russia	.....	.....	88,000
Other countries	.....	2,100,000	.....
Total	79,738,000	97,000,000	141,414,000

TABLE NO. XV

Output of Domestic Rayon Manufacturers

(in pounds)

		1925	
	1924	First Six Months	Last Six Months
Viscose	28,000,000	17,000,000	19,000,000
Du Pont	4,000,000	3,000,000	4,500,000
Tubize	4,250,000	2,600,000	2,600,000
Industrial	2,000,000	1,250,000	1,750,000
Celanese (Acetate)	.....	500,000	1,000,000
Lustron (Acetate)	500,000	250,000	250,000
Bemberg	.....	.....	.....
Belamose	.....	*175,000	500,000
Acme	250,000	150,000	175,000
Total	39,000,000	24,925,000	29,775,000

	1925	1926
Viscose	36,000,000	45,000,000
Du Pont	7,500,000	*20,000,000
Tubize	5,200,000	6,000,000
Industrial	3,000,000	3,500,000
Celanese (Acetate)	1,500,000	3,500,000
Lustron (Acetate)	500,000	500,000
Bemberg	.....	1,000,000
Belamose	675,000	*1,000,000
Acme	325,000	600,000
Total	54,700,000	*81,100,000
Per cent of total production as acetate	3.7	4.9

*Changed to include the new unit in Buffalo, N. Y.

The Preparation and Constitution of Cellulose Acetate

In order that we may have a better understanding of the whole subject, not only of dyeing acetate silk, but of its manufacturing operations, the causes of variations, differences, characteristics, etc., it may be well to give a few details regarding the preparation of cellulose acetate, its chemical constitution, physical and chemical properties, general manufacturing operations of acetate silk, etc.

Professor Heuser in "Cellulose Chemistry," states that Schnutzenberger prepared cellulose acetate in 1866 from cellulose and acetic anhydride by heating them in a sealed tube. Later experiments were made by Franchimont in 1879 and by Skraup. Cross and Bevan in 1901 were the first to give it a technical value. Bayer possibly made the first attempt to utilize it commercially about 1904.

Heuser states that cellulose acetate is formed according to the equation: $C_6H_{10}O_5 + 3(CH_3CO)_2O = C_6H_7O_5(CH_3CO)_3 + 3(CH_3COOH)$.

Theoretically, according to this equation, 100 grams of air-dry cellulose, such as cotton with 5 to 6 per cent of water, will require 180 grams of acetic anhydride. However, in practice an excess of the anhydride, or about 400 grams per 100 grams of cellulose are used.

Ost, in the *Zeit. f. angew. Chem. 32*, 66-70, 76-9 and 82-9 (1919), divides the cellulose acetates into two classes: (1) primary products of the acetylation of cellulose or hydrocellulose, which are soluble in chloroform, preferably with the addition of a little alcohol; and (2) secondary acetates derived from the primary acetates by mild processes of hydrolysis or saponification. The primary acetates show a partial and fugitive solubility in acetone, and while their films from chloroform are good, those from acetone are poor and can only be partially redissolved by acetone. On the other hand the acetone films from the secondary products are good and complete solubility in acetone is a permanent characteristic. He also divided the "catalysts" into two groups: (1) those containing no sulphuric acid, of which he studied only zinc chloride; and (2) sulphuric acid or dissociable compounds capable of liberating sulphuric acid, such as the sulphates of organic bases.

He states that a "pure cellulose triacetate" may be obtained by treating 100 parts of cellulose, purified by treatment with dilute caustic soda and containing 5 to 6 per cent of moisture, with 400 to 500 parts of acetic anhydride, 400 to 500 parts of glacial acetic acid and 50 to 100 parts of fused zinc chloride. After about three weeks' time at 20 deg. Cent. (68 deg. Fahr.) the triacetate is obtained containing 62.5 per cent of acetic acid. At 10 or 12 deg. Cent. (50 or 54 deg. Fahr.) several months' treatment is required to complete the reaction. Higher temperatures (60 to 70 deg. Cent.) shorten the period of esterification, but cause considerable breaking down of the molecule and the product is of little value. The triacetate formed in this manner dissolves in pure chloroform, but is more soluble in the presence of some alcohol, in which case viscous solutions may be obtained. As a primary acetate it is not soluble in acetone, as will be explained later.

When cotton is immersed in a mixture of 50 parts of acetic anhydride, 50 parts of glacial acetic acid and 4 to 6 parts of sulphuric acid at 30 to 40 deg. (86 to 104 deg. Fahr.) it rapidly dissolves to form:

$$(C_6H_7O)_4SO_4(C_2H_3O_2)_{10}.$$

Thus, like the nitration of cellulose, the acetylation is incomplete in the presence of sulphuric acid. The acetic acid content in this case, if cleavage of the molecule is avoided, is only 59 to 61 per cent and sulphoacetate is formed simultaneously, the quantity depending upon the amount of sulphuric acid used as "catalyst." Pure high molecular acetates, free from sulphoacetates, cannot be prepared with sulphuric acid as catalyst. At a temperature of 10 to 15 deg. Cent. (50 to 59 deg. Fahr.), two to four days' acetylation in the presence of 5 or 10 per cent of sulphuric acid gives the best result. Prolonged acetylation at low temperatures appears to increase the amount of combined acetic acid in the product, with a consequent increase in the amount of combined sulphuric acid. With increasing time and temperature, the sulphuric acid causes a greater acetolytic decomposition of the cellulose molecule. When only 1 to 2 per cent of sulphuric acid is used, the acetylation is complete in a few hours at 35 to 40 deg. Cent. (95 to 104 deg. Fahr.).

When used as "catalysts," acids combined with weak bases are far more moderate in their action on the product than sulphuric acid. For instance, when methylamine sulphate is used as catalyst, temperatures may be increased to 35 or even 50 deg. Cent. With 5 to 10 per cent of methylamine sulphate at 35 deg. Cent., several days are necessary for the complete acetylation, but the molecular cleavage is slight even in ten days. At 65 to 70 deg. Cent. the danger is increased considerably, but the acetylation is complete in two hours. The composition of this methylamine sulphate acetate is similar to that obtained with sulphuric acid but it contains less sulphoacetate. According to Ost, pure acetates of low acetic acid content are difficult to obtain as primary acetates. If insufficient acetic anhydride is present to form the triacetate, a portion of the cellulose remains unaltered.

while the composition of the acetate formed approaches that of the triacetate. Insufficient time or a catalyst (zinc chloride) affect the final product in the same manner.

Unsaturated esters are readily obtained with either an excess or a deficiency of acetic anhydride if a large proportion of sulphuric acid or methylamine sulphate is used. The acetic acid content of the final cellulose acetate may fall to 48.5 per cent (diacetate), and even below 40 per cent, but with a simultaneous rise of combined sulphuric acid of from 2.3 to 7 per cent. These mixed esters are partially hydrolyzed on precipitation with water; are increasingly insoluble in chloroform, in which they swell, but dissolve with inclreasing sulphuric acid content in alcohol and finally in water. While pure cellulose triacetate may be preserved for years and is not affected by boiling water, products containing sulphoacetates split off sulphuric acid or boiling in water.

W. Weltzein and R. Singer, *Annalen 443*, 71-112 (1925), state that the action of acetic anhydride, in the presence of sulphuric acid, on cellulose leads to the production of cellulose acetate, cellulose acetate A, cellodextrin acetates which are divisible into a number of groups in accordance with their solubility or insolubility in alcohol and ether, cellobiose octa-acetate, and dextrose penta-acetate.

It is interesting to note that Caille, *Chem. et Ind. 13*, 11-13 states that acetate silk which contains combined sulphuric acid resists hydrolysis or heating with water at 120 deg. Cent. (248 deg. Fahr.), if the sulphuric acid groups have been neutralized by washing with calcareous water. Any unneutralized portion of such groups readily splits off by heating with water under pressure.

According to Ost only a portion of all primary cellulose acetate is extracted by pure acetone, this extraction being least in the case of a zinc chloride triacetate and most in the case of a methylamine sulphate product, often amounting to from 50 to 80 per cent of its weight. While the chloroform solubility is constant, the acetone solubility is not. An acetone film is only partly soluble in acetone a second or third time. None of the primary triacetate is soluble in acetone, but only secondary acetates which result from partial hydrolysis. Ten per cent of aqueous mineral acid at 20 deg. Cent., or 1 per cent at 100 deg. Cent. (212 deg. Fahr.) produces hydrolysis of the triacetate but does not give an acetone soluble product. Real and permanent acetone solubility is produced by partial hydrolysis of primary acetates of low acetic acid content and high sulphuric acid content, by stirring a little acid water into the finished acetylated syrup. See German Patent No. 252,706, which follows.

Exceptional acetone solubility results by heating a primary acetate or the primary syrup, after neutralization of the sulphuric acid, with 95 per cent acetic acid for some time. Hydrolysis takes place in from 24 to 96 hours to a combined acetic acid content of from 56.2 to 50.8 per cent. Heating in the presence of small quantities of catalysts, such as sodium bisulphate, gives similar results. Heating with aniline or phenol, in the presence of a little water, also hydrolizes it to the acetone-soluble form. Solubility in acetone is not connected with any definite degree of hydrolysis, the limits of the combined acetic acid varying betkeen 57.6 and 50.9 per cent or even more widely.

Primary acetates prepared from previously hydrolized cellulose are very similar to those prepared from ordinary cellulose. The resulting films appear to be just as good but generally the solutions are of lower viscosity and the precipitated products are more pulverulent. Hydrocellulose is more readily acetylated than ordinary cellulose and the reaction proceeds more rapidly, with a smaller quantity of catalyst and at a lower temperature. Secondary acetates are produced from these products in the same manner as from those prepared from cellulose.

Barnett, *J. Soc., Chem. Ind. 40*, 8 (1921), prepared cellulose acetate from filter paper by pouring 200 grams of glacial acetic acid, containing just sufficient chlorine to color it, on 50 grams of the paper. After standing some time, 250 grams of acetic anhydride were added and a few bubbles of sulphur dioxide passed through the mixture. The temperature at once increased and when maintained at 65 deg. Cent. (149 deg. Fahr.) by cooling, a clear solution of cellulose di-acetate was obtained in about an hour. This product was soluble in chloroform, acetone, pyridine, aniline, hot nitrobenzene and slightly soluble in a mixture of benzene and alcohol. In another experiment 5 grams of filter paper was immersed in 20 c.c. of glacial acetic acid, and 20 c.c. of acetic anhydride containing 0.32 gram of chlorine added. Then 2 c.c. of acetic anhydride containing 0.26 gram of sulphur dioxide were further added, whereby the mixture rapidly gelatinized and solution was complete within five minutes. White flakes of cellulose triacetate were separated from the product. Also see the discussion under "Catalysts."

German Patent No. 252,706, to Bayer, specifies 15 per cent of sulphuric acid and a limited amount of acetic anhydride in the primary acetylation. The product resulting from one day's acetylation may contain over 3 per cent of combined sulphuric acid, but if a longer time is allowed, the combined acetic acid increases, with a decrease in the sulphuric acid content. However, a breakdown of the cellulose occurs simultaneously with this change in composition. The patent suggests the addition of a little water to the primary syrup and it is then allowed to "ripen." With this modification there is less breaking down of the cellulose and the viscosity of the syrup is maintained while the combined acetic and sulphuric acid content decreases, the latter falling to 0.25 per cent in one or two days, and to 0.17 per cent after four days. The acetic acid values showed 57.4 per cent after one day, 54.0 per cent after two days, and 50.9 per cent after four days at 20 to 23 deg. Cent. These products are soluble in acetone and give good films.

According to British Patent No. 139,232 (1918) to J. O. Zdanowich a good grade of cellulose acetate, suitable for the manufacture of acetate silk, may be prepared by treating 150 grams of purified cellulose with a mixture of 500 c.c. of glacial acetic acid and 420 c.c. of acetic anhydride. While agitating, chlorine gas is passed into

the mixture at 70 to 80 deg. Cent. (158 to 176 deg. Fahr.). After two or three hours, 2 grams of sulphuric acid are added and stirring continued until a clear solution is obtained. This solution is generally precipitated by water, washed, dried and dissolved in the spinning solvent, usually acetone. It is understood that this solution of cellulose acetate is forced through fine jets, either into a spinning solution or more frequently into warm air, in about the same general manner as in the manufacture of the other rayons.

"Catalysts"

Professor Heuser, *loc. cit.*, discusses the various "catalysts" used in the manufacture of cellulose acetate. Very evidently, from the quantity of these materials required in the acetylation process, their role is not strictly that of a catalyst. While they do start and accelerate the reaction, their part is possibly principally that of a peptonizing agent, rather than that of a catalyst. These "catalysts" no doubt act on the cellulose in such a manner as to render it more reactive with the acetic anhydride, and while in most instances their action on the cellulose is not great, the resulting acetate is not a pure cellulose acetate when they are used. Acetic acid acts as a solvent for the reaction as well as the acetate formed, but alone it has very little esterifying action on cellulose even at high temperatures.

Barnett, *loc. cit.*, in discussing the catalysts, says that most of them are strong dehydrating agents and as examples mentions sulphuric acid, phosphoric acid, zinc chloride, chlorides and oxychlorides of sulphur and phosphorus, dimthyl sulphate, chloroacetic acid, etc., chlorine does not react readily except under special conditions and then only slowly. Sulphuric acid tends to give dark colored solutions and to hydrolize the product. It may also cause brittleness in the film, due to its incomplete removal. Sulphuryl chloride (see British Patent No. 24,382 of 1910), is claimed to give a superior product. Either chlorine or sulphur dioxide alone give poor results as catalysts, but when traces of both are introduced either separately or together, the acetylation proceeds to a remarkable extent giving a perfectly clear solution or jelly. This catalyst is also useful in the preparation of other cellulose esters, such as the butyrate, benzoate (using benzoyl chloride in acetic acid), etc. He attributes the great reactivity of this mixed catalyst to a possible condensation of the chlorine and sulphur dioxide upon the cellulose itself, which produces a small amount of sulphuryl chloride *in statu nascendi* in intimate contact with the cellulose. The reaction proceeds best when the amounts of chlorine and sulphur dioxide are approximately in the ratio of their molecular weights.

Besides the solvents given on page 174 of Part I, cellulose acetate is soluble in various organic solvents such as tetrachloracetylene and benzyl alcohol. Cyclohexanone and other cyclic ketones of the same series or their alkyl or alkyl substitution products have been suggested also as solvents and patents have been issued covering their use for this purpose. Chlorohydrin and dichlorohydrin have been patented as solvents for the higher acetylated products (50 per cent acetic acid), and hot nitrobenzene and aniline have also been mentioned.

From the X-ray examination of fibrous materials, Herzog and Jancke, *Umschau 25*, 53-4 (1921), report that cellulose and true silk are crystalline in structure and that the usual rayon, except acetate, consists of an irregular felted mass of crystalline fragments of unchanged cellulose. Acetate silk is amorphous and consists of a mixture in which the cellulose acetate has undergone considerable degradation (hydrolysis?).

Identification

Professor Johnson's paper in the American Dyestuff Reporter, *14*, 105-7 (1925) upon the identification of acetate and other artificial silks is the most complete article which has appeared upon the subject, but as the original is available to the readers of this journal, it is not necessary to discuss it here. Some other tests recently announced by the Bureau of Standards are reported in American Dyestuff Reporter *14*, 404 (1925).

Before making the colorimetric tests upon dyed rayon, it is usually necessary to "strip" the color from the fiber. This may be done by the usual methods with hypochlorite, peroxide, permanganate, hydrosulphite or a hydrosulphite-formaldehyde compound, such as Protolin AZ, in a solution containing acetic or formic acid. The latter method is recommended, but will not remove all dyes from acetate silk.

The Ruthenium Red Test

Dr. John A. Haerry in "Artificial Silks" states that ruthenium red, a complex salt of ruthenium oxychloride with ammonia, $Ru_2(OH)_2Cl_4(NH_3)_7.3H_2O$, is useful as a reagent in the identification of the rayons. According to C. F. Green, Textile Colorist 47, 177-8 (1925), this reagent colors lignin, pectin material and oxycellulose. Upon treating rayon with a one-tenth of one per cent aqueous solution of this reagent and allowing it to stand twelve hours, viscose becomes a distinct pink, more pronounced on standing; denitrated Chardonnet rayon first becomes red, but is more violet on standing; cupra rayon is only slightly pink even after standing twelve hours; acetate silk which should be uncolored is usually irregularly tinted after twelve hours; and rayon which has received and formaldehydestenose treatment is uncolored. This irregular tinting of acetate silk indicates irregularity in its composition and this test might be used for uniformity. Naphthylamine Black 4B (Cassella) dyes copper rayon dark blue and viscose a light blue from a hot neutral dye bath. Acetate rayon dissolves completely in acetone or in glacial acetic acid, while the other varieties do not.

The Iodine-Zinc Chloride Test

This reagent is useful in identifying the various rayons. It is prepared by dissolving (*a*) 20 parts of zinc chloride in 10 parts of water; and (*b*) 2 parts of potassium iodide and 0.1 part of iodine in 5 parts of water. Solutions *a* and *b* are mixed, allowed to settle, and a few crystals of iodine added to the clear solution. With this reagent acetate silk is colored a distinct yellow; cupra a brownish tint which is easily washed out; nitro a reddish-violet; and viscose a bluish-green which is not fast to washing.

Upon heating small samples of rayon in Fehling's solution on the water bath, cupra and viscose rayons leave the solution a blue color, while the nitro rayon changes it to a greener shade. The fact that the luster of acetate silk is impaired by hot caustic solutions has also been used as a quick test for this fiber. The other rayons withstand this test better than the acetate. They all swell to some extent, but none dissolve, so that this test will readily serve to distinguish the rayons from the true silk. Glycerol-copper sulphate reagent may also be used to distinguish between rayon and real silk. It is prepared by dissolving 2 parts of copper sulphate in 20 parts of water, adding one part of glycerol and then sufficient concentrated caustic potash solution to redissolve the precipitate which is at first formed.

Another method suggested for identifying nitro rayon is to dissolve the sample in chemically concentrated sulphuric acid and add brucine sulphate to this solution. When the sample contains nitro rayon, the solution becomes a bright red color. It has also been stated that Celanese will dye a deeper shade with the SRA dyes than Lustron, while none of the other rayons are dyed by these products. Acetate silk is not soluble in ammoniacal-cupric oxide reagent, while the other rayons dissolve slowly. This reagent is prepared by dissolving copper sulphate in water and precipitating the copper oxide with ammonia. This precipitate is washed and dissolved in concentrated ammonia. Acetate silk swells in chromic acid solution but is not dissolved, while the other rayons are dissolved.

The Acetate Test

The "acetate test" has frequently been recommended as a test for acetate silk. Possibly the best method of making this test is to dissolve the fiber in a little chloroform and boil this with a normal solution of sodium hydroxide. Dilute with water, filter off the precipitated cellulose and evaporate the solution just to dryness. Add sufficient water to dissolve the residue and acidify with sulphuric acid. Add a little ethyl alcohol and the same amount of sulphuric acid, warm, and if the sample contained acetate silk, the odor of ethyl acetate may be detected.

Acetate Silk Saponified in Scouring

The sensitiveness of acetate silk to hot alkaline solutions is a frequent cause of unevenness in dyeing. With basic dyes the uninjured portions of the fiber are colored darker than the saponified portions. Practically all of the special acetate silk dyes act in the same manner. With dyes which color cotton, the reverse is the case, i. e., the hydrolyzed portion of the fiber is colored the most. This fact serves as the basis of a quick test for the detection of acetate silk which has been injured in scouring. Simply dye a sample of the suspected acetate silk and a properly scoured sample, with a dyestuff which does not stain acetate silk, such as those given in Part XIV. The colored portions of the skein have been injured by alkali.

It has been stated that where unevenness in dyeing is caused by variations in the constitution of the acetate silk itself, more level results are usually obtained with the older dyestuffs which are applied direct, such as the acid and basic dyes, than with the special acetate silk dyes, such as those of the dispersal class.

Regain and Strength

Professor Grimshaw in Textile World *67*, 1397-9 (1925), reports experiments which show that when Celanese is exposed to an atmospheric relative humidity of 84 per cent, the equilibrium regain is only 8.59 per cent, while under the same conditions, cotton has 122.5 per cent of regain and viscose 15.69 per cent. Under room conditions, wherein cotton had 6.8 per cent of regain and viscose 9.52 per cent, the Celanese had only 3.57 per cent of regain. In other words, it usually contains only about one-third to one-half as much moisture as the other rayons. He also gives Table No. XVI showing the strength of Celanese as compared with viscose (Du Pont) rayon.

Table No. XVII is given by Wilson of Courtaulds, and Tables No. XVIII, XIX and XX by W. F. Edwards, Textile World *68*, 1792-4 (1925).

TABLE NO. XVI

Strength of 150-Denier Du Pont Viscose and Celanese Rayons in 120-Yard-Length Skeins on Scott Tester

Moisture corresponding to cotton regain of—	Break per Skein	
	Du Pont Vis.	Celanese
6.7 per cent	59.3 pounds	58.7 pounds
7.7 per cent	60.0 pounds	61.0 pounds
12.5 per cent	55.3 pounds	49.3 pounds
13.7 per cent	55.0 pounds	49.7 pounds

TABLE NO. XVII

Comparative Strengths of Various Rayons

	Tensile Strength		
	Dry	Wet	Extension
Chardonnet	0.75-1.40	0.25-0.60	7.5-16.0%
Cuprammonium	1.00-1.35	0.35-0.55	14.0-18.0%
Acetate	1.1	0.7	18.0%
Viscose	1.20-1.60	0.45-0.70	11.0-22.0%

TABLE NO. XVIII

Per Cent Moisture in Different Varieties of Rayon

Temperature °C.	°F.	Relative Humidity	Raw Silk	Nitro-Rayon, Proc. A	Nitro-Rayon, Proc. B	Acetate Rayon*
14.5	58.....	62%	9.834	10.150	10.346	5.134
14.5	58.....	72%	10.647	10.729	10.862	5.626
20.0	68.....	60%	9.460	9.956	9.457	4.600
18.0	64.....	65%	9.834	10.248	9.773	4.921
19.0	66.....	65%	7.148	10.334	9.825	4.957
18.5	65.....	72%	8.319	11.016	10.605	5.556
18.5	65.....	72%	8.626	11.158	10.708	5.591
20.5	69.....	70%	8.809	11.068	10.708	5.486
19.0	66.....	73%	8.931	11.347	10.913	5.696
20.0	68.....	75%	9.007	11.536	11.117	5.905

*Celanese.

TABLE NO. XIX

Lower Limit of Strength and Stretch for the Best Grades of Raw Silk and Types of Rayons

	*Strength	†Stretch
Raw silk	3.50 up	20 up
Regen. rayon from cotton linters	1.70 up	20 up
Regenerated rayon from wood pulp	1.50 up	20 up
Acetate rayon	1.30 up	30 up

*In grams per denier. †Per cent elongation.

FILAMENT SIZES OF RAYONS

Chittick in Textile World *68*, 1776 (1925) gives data regarding the denier and number of filaments in the different varieties of rayons:

TABLE NO. XXI

Tubize or Nitro Cellulose Rayon

Denier Size	No. of Filaments	Deniers per Filament
50	10	5
60	10	6
70	10	7
80	10	8
90	12	7 1-2
100	12	8 1-3
110	12	9 1-6
120	20	6
130	20	6 1-2
140	20	7
150	20	7 1-2
160	20	8
170	20	8 1-2
180	20	9
Viscose Rayon		
120	14	8 4-7
130	14	9 2-7
150	18	8 1-3
300	28	10 5-7
450	42	10 5-7
Elberfeld Cuprammonium Rayon		
250	18	13 8-9
Celanese (Acetate) Rayon		
45	13	3 3-16
65	20	3 1-4
90	30	3
130	40	3 1-4
150	26	5 10-13
180	60	3
300	50	6
Lustron (Acetate) Rayon		
150	16	9 3-8
Bemberg Viscose Rayon		
120	(stated) 90	1 33
150	(stated) 110	1.36

MERCERIZING CELANESE UNIONS

Welch in a recent paper states that Celanese is able to withstand the mercerizing operation in unions and that it is actually being done in at least two plants. He also states that the luster which has been destroyed on Celanese by high temperature can be restored by a very simple process (not given), provided that the fiber has not been

(*Continued on page 765*)

TABLE NO. XX

Strength and Stretch of Various Rayons

	Number of Threads	Denier Size	Strength, Gms. per Denier	Stretch, Per Cent Elongation
Regenerated cellulose rayon from wood pulp:				
American I	100	150	1.56	27.5
American 55	200	150	1.54	28.5
(Same grade, A, from same factory, No. 1)				
American III; factory No. 2, best grade A	200	150	1.60	24.5
American IV; factory No. 3, best grade A	200	150	1.56	23.0
European; grade not known	200	150	1.48	20.0
European; grade C	100	120	1.42	23.0
Regenerated cellulose rayon from cotton linters:				
European; grade not known	200	80	1.63	21.5
European; grade not known	100	300	1.56	20.0
American; factory No. 1, best grades	200	150	1.76	20.0
American; factory No. 2, best grades	200	150	1.85	22.0
Acetate rayon:				
European	200	150	1.30	29.0
European	50	300	1.20	36.0

The Reduction Products of Azo Dyes

Part IV

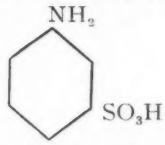
By WALTER C. HOLMES

Color Laboratory, Bureau of Chemistry

[EDITOR'S NOTE.—*The most practical and effective method for the identification of azo dyes consists in the isolation and identification of the products which they yield on reduction. This method finds less application than it might or should, partly because some of these products have never been isolated and investigated, and partly because much of the data which has been obtained in regard to such reduction products is greatly scattered and very difficult to locate.*

Mr. Holmes has made a search for all the available information on such reduction products, and this series contains a digest of his results with a systematic listing of the reduction products of the azo dyes published in the Colour Index, together with the available data on such properties and reactions as would be of service in their identification.]

No. 30.—METANILIC ACID



$C_6H_7NO_3S$; Mol. Wt. 173.15; (C, 41.6%; H, 4.1%; N, 8.1%; O, 27.7%; S, 18.5%).

Derived from: C. I. Nos. 138 (139?, 140?), 141, 233, 289 and 290.

Long colorless needles or prisms ($+1\frac{1}{2}H_2O$).

Hydrochloride, needles, mpt. 235° (decomp.), readily sol. in H_2O and alc. Metallic salts are readily sol. The *Ba salt* gives no pptte. with bromine water (distinction from o- and p- isomers).

Acetyl deriv., the free acid is not acetylated.

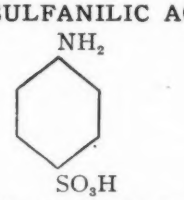
Na acet-metanilate ($+2H_2O$), long colorless needles, loses H_2O at 100° and melts at 184-5°; *Ba acet-metanilate* ($+2H_2O$), glittering colorless needles, decomp. over 300°.

Sulfonamide, glistening leaflets or long needles, mpt. 142°, difficulty sol. in cold H_2O, readily in hot; *acetyl sulfonamide,* small rhombs (Ac HO), mpt. 216-9°, sol. in acetone or hot Ac HO; *chloracetyl sulfonamide,* needles (alc.), melts incompletely at 153-5° and gives clear melt at 165°.

Gives m-diazobenzene sulfonic acid with nitrous acid.

Iodo derivs., see J. Chem. Soc. *95,* 1683.

No. 31.—SULFANILIC ACID



$C_6H_7NO_3S$; Mol. Wt. 173.15; (C, 41.6%; H, 4.1%; N, 8.1%; O, 27.7%; S, 18.5%).

Derived from: C. I. Nos. 16, 142-155, 234, 238, 239, 241, 273-282, 291-295, 322, 323, 349A, 585, 598, 599, 606, 610, 611, 639 and 640.

Rhomb. plates. Dihydrate below 21° (efflorescent), monohydrate 21°-40°, anhydrous over 40°. Sol. in 224 parts H_2O at 0°, 90 parts at 18.9° and 34 parts at 54.5°.

The hydrochloride and metallic salts are readily sol. The Ba salt gives a pptte. of tribromaniline, mpt. 119°, with bromine water (distinction from metanilic acid).

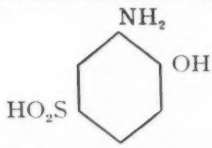
Acetyl-sulfanilic acid—Chloride, small needles (C_6H_6), mpt. 149°, sol. in alc., ether and Ac HO, insol. in H_2O; *—ethyl ester,* white needles (C_6H_6), mpt. 115°;—*p-phenetidine,* white glistening leaflets, mpt. 204°. *Benzoyl-sulfanilic acid chloride,* yellow needles (C_6H_6), mpt. 176°. *Benzolsulfon-sulfanilic acid,* mpt. 178°, readily sol. in H_2O;—*chloride,* white needles (Ac HO), mpt. 177°.

Gives p-diazobenzene sulfonic acid with nitrous acid.

Gives quinone with chromic acid mixture (distinction from metanilic acid).

With ferricyanide it gives a flesh-red azoxy dye, easily sol. in warm water with a yellow color.

No. 32.—2-AMINOPHENOL-4-SULFONIC ACID



$C_6H_7NO_4S$; Mol. Wt. 189.15; (C, 38.1%; H, 3.7%; N, 7.4%; O, 33.8%; S, 16.9%).

Derived from: C. I. Nos. 167-170.

Colorless rhombohedra or monocl. prisms ($+\frac{1}{2}H_2O$). Sol. in about 100 parts H_2O at 14°. Somewhat unstable in boiling H_2O. Decomposes without melting.

Does not form salts with acids. Alkali salts readily sol; *Ba salt,* difficultly sol. in H_2O and alc., colorless if pure; *Cu salt,* clear green needles; *Co salt,* red pptte.

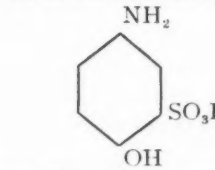
Na salt of benzoyl deriv. ($+4\frac{1}{2}H_2O$), colorless (if pure), easily sol. in H_2O and alc.

Anilide ($C_6H_3.OH.NH_2.SO_2NHC_6H_5$), needles, mpt. 205°, insol. in ether or ligroin, readily sol. in alc., C_6H_6 and Ac HO.

Gives a yellow diazo deriv. ($+H_2O$, lost at 80°), the color being practically unaffected by alkalies or acids.

Polyazo deriv.—see D. R. P. 45994, Friedl. II, 319.

No. 33.—4-AMINOPHENOL-2-SULFONIC ACID



$C_6H_7NO_4S$; Mol. Wt. 189.15; (C, 38.1%; H, 3.7%; N, 7.4%; O, 33.8%; S, 16.9%).

Derived from: C. I. No. 408.

Long needles, hexagonal plates or large wedge-shaped cryst. Cryst. with 1 mol. H_2O (lost at 100°). Stable if sl. acid but turns brown in moist air, in presence of NH_3

and on heating. Decomp. without melting. Sol. in about 1,500 parts H_2O at 14°, insol. in alc. and ether. Reduces ammon. $AgNO_3$ in cold. Is colored violet by $FeCl_3$. Not pptted. by lead acetate.

Does not form salts with acids. The metallic salts are not particularly characteristic (including those of the benzoyl deriv.).

Anilide, colorless needles (alc. + H_2O) or small dense cryst. (Ac HO + H_2O), mpt. 98°.

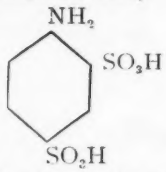
On dry distil. (unlike the o-amino isomer) it gives a dark violet dye ($C_{18}H_{15}NO_3$), cobalt-blue sol. with alkalies.

Gives a colorless diazo deriv. (+H_2O) which turns yellow with alkalies and gives a pale yellow brucine salt.

If a suspension in Ac HO is treated with bromine a yellow pptte. is obtained of *bromanil* ($C_6Br_4O_2$), which sublimes at about 220° and melts at about 300°.

By heating with HCl to 180° it gives p-aminophenol hydrochloride (base, mpt. 183°).

No. 34.—ANILINE-2, 4-DISULFONIC ACID (Disulfanilic Acid)



$C_6H_7NO_6S_2$; Mol. Wt. 253.21; (C, 28.5%; H, 2.8%; O, 37.9%; S, 25.3%).

Derived from: C. I. No. 296.

Brownish-red warts of microse. cryst. (2 or 2½ H_2O). Readily sol. in H_2O and alc., insol. in ether. Decomp. above 120°.

Gives mono- and di-basic metallic salts, the latter being more sol.

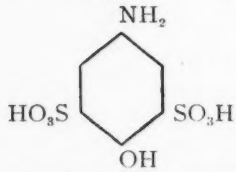
Chloride, mpt. 63°.

Amide, glistening leaflets (H_2O), mpt. 235° (Fischer); mpt. 229° (Limpricht).

With bromine water gives tribromaniline, mpt. 118.5°.

Heated with alc. under pressure gives m-disulfobenzene which gives a cryst. chloride, mpt. 61°.

No. 35.—4-AMINOPHENOL-2, 6-DISULFONIC ACID



$C_6H_7NO_7S_2$; Mol. Wt. 269.21; (C, 26.8%; H, 2.6%; N, 5.2%; O, 41.6%; S, 23.8%).

Derived from: C. I. No. 171.

White, silky, deliquescent needles, difficultly sol. in alc., insol. in ether.

The acid alkali salts are difficultly sol. in H_2O, the neutral lead salt nearly insol.

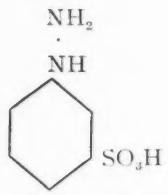
Sols. of the acid and its salts give a violet color with $FeCl_3$.

Dilute alkaline salt sols. show a characteristic blue fluorescence which is transitory (with alkali carbonates this is replaced soon by a green color).

Gives a yellow diazo deriv., small needles, sol. in H_2O, insol. in alc.

For characteristics of isomeric acid, see D. R. P. 65236 (Friedl., III, 56).

No. 36.—PHENYLHYDRAZINE-m-SULFONIC ACID



$C_6H_8N_2O_3S$; Mol. Wt. 188.17; (C, 38.3%; H, 4.3%; N, 14.9%; O, 25.5%; S, 17.0%).

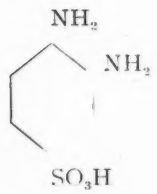
Derived from: C. I. No. 367 by partial reduction.

Rhomb. plates or needles. (½H_2O) Sp. sol. in cold H_2O, readily in hot H_2O, practically insol. in alc. and ether.

With nitrous acid gives triazobenzenesulfonic acid.

Gives metanilic acid on reduction.

No. 37.—o-PHENYLENEDIAMINE-p-SULFONIC ACID



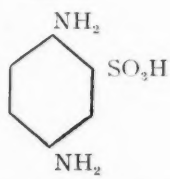
$C_6H_8N_2O_3S$; Mol. Wt. 188.17; (C, 38.3%; H, 4.3%; N, 14.9%; O, 25.5%; S, 17.0%).

Derived from: C. I. No. 159 by complete reduction.

Colorless cryst. which turn blue-green in air. Very difficultly sol. in cold H_2O, easily in boiling H_2O. Its sol. is colored an intense brown by lignin and red-brown by $FeCl_3$.

Forms salts with both bases and acids. *Na salt* cryst. with 1H_2O. *Hydrochloride*, fine colorless, difficultly sol. needles. It is unstable, losing HCl on repeated cryst. from hot H_2O.

No. 38.—p-PHENYLENEDIAMINESULFONIC ACID



$C_6H_8N_2O_3S$; Mol. Wt. 188.17; (C, 38.3%; H, 4.3%; N, 14.9%; O, 25.5%; S, 17.0%).

Derived from: C. I. Nos. 16 and 279-282 and by the complete reduction of 157, 158 and 541.

White needles (+2H_2O), which soon turn greenish in air. Moderately sol. in H_2O, nearly insol. in alc., insol. in ether and C_6H_6. Readily sol. in HCl.

Na Salt (+4H_2O), dense cryst., easily sol.

Gives the indamine reaction in presence of m-toluylenediamine and neutral $FeCl_3$ and indophenol formation in presence of sodium-α-naphthol.

Oxidized with warm alkaline $K_3Fe(CN)_6$ it gives a chocolate colored azoxy dye, sol. in hot H_2O with a red color, which dyes wool blue-green.

(*Continued on page* 776)

Proceedings of the American Association of Textile Chemists and Colorists

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OCTOBER MEETING OF THE NEW YORK SECTION

The regular monthly meeting of the New York Section of the American Association of Textile Chemists and Colorists convened at the Machinery Club, 50 Church Street, New York City, at 8.15 P. M., October 23.

After the secretary had read the minutes of the last meeting and the treasurer had presented his report the chairman introduced the principal speaker for the evening.

Chairman Grandage—Gentlemen, we have with us tonight Dr. Lesser of the American Dyewood Company who is going to present a paper on the subject of "Some Applications of Logwood." After our last meeting, a number of men came to me and asked if we could arrange to have Dr. Lesser present this subject at as early a date as possible. I am quite sure that after he presents his paper, Dr. Lesser will be glad to answer any questions that he is able to, although he informs me that he prefers to have the questions asked this month and answered next month. However, I think he will take care of you all right.

Some Applications of Logwood

By DR. EMIL LESSER
American Dyewood Company

Mr. Chairman and Gentlemen: It is only a few days since my old friend Mr. Knowland asked me to give this little talk. As far as I know, I have to-day become a member of this Society. I did not know that a newly elected member would have to give a speech. (Laughter.)

I do not intend to give you a long talk about the great value of Logwood, nor do I intend to make comparisons with the so-called synthetic dyestuffs. I shall confine myself to some practical applications of Logwood, applications with which I am familiar through my own experiences as demonstrator for the American Dyewood Company, or through the experience of other men. I shall not go into any great discussions about theories. But I think it is necessary before talking about the practical use of Logwood that I give you some information about it.

I would like to put before you, first of all, the question: What is Logwood?

The dye substance which is known in the trade as Logwood extract or Hematin is made from the wood of a tree which originally was a native of Mexico and Central America. From there the tree was transplanted to other countries, especially the West Indies.

It is interesting to observe that environment, climate, soil, and so forth, affect the plant the same as they do a human being. The tree now growing in Jamaica was transplanted there from Honduras in about 1715. The Honduras wood of to-day and the Jamaica wood show marked differences, both physically and chemically. The Honduras wood has a round section, whereas the Jamaica wood is very irregular and full of grooves and gutters. Besides, the extract of a Honduras wood will give quite different results when used on cotton than the Jamaica wood will give.

The reason I am explaining these things to you is to

show that it is necessary to select a certain Logwood for each different kind of material.

Also we must take into consideration the fact that the making of Logwood extract depends a great deal on the origin of the wood. The wood itself, it might interest you to know, grows in a warm, moist climate. The Logwood does not require a special soil. There is no attempt made to cultivate it. It is a self-sown tree; in fact, it is only necessary to thin out the saplings in order to give promising trees advantage to grow; otherwise a Logwood plantation would become a veritable jungle.

When the tree is about eight to twelve years old, it is marketable. That means it contains enough of a certain substance—a glucoside—which will give the coloring matter to the extract. The trees are then felled and cleaned of the smaller branches, partly of the bark, and sent to the United States and Europe. Sometimes even the roots are used, and give valuable dyestuff. The wood is then cut up into small chips by a machine which is called the wood-hog.

After the wood is cut into chips, it is treated in big extractors with steam and water. The American custom is to use the so-called closed extractors. On the other hand, the people in Europe, especially the French, use so-called open extraction apparatus. There is in the wood a certain body which yields the coloring matter to the extract. We know little about this body, only that it is a glucoside. This latter produces a body which we call Hematoxylin, the principal portion of the Logwood extract. When the Hematoxylin is oxidized either by the air or by oxidizing agents it is transformed into Hematin, the real coloring matter of the Logwood.

We have, therefore, two different kinds of products, one called Logwood extract, consisting mostly of Hematoxylin and about 15 to 20 per cent Hematin, and the other called Hematin extract consisting mostly of Hematin, about 60 to 80 per cent generally. These two different extracts are found in the trade. When evaporated to dryness under certain precautions these extracts yield the so-called Logwood, respectively, Hematin crystals. Logwood extract comes on the market also in solid form as a block in boxes.

You must bear in mind that Logwood is an adjective dye. As you all know, that means it requires for the fastening and developing of color on the fiber (or whatever the material is) something which is known in dye literature and in the dyeing art as a mordant. I am speaking rather plainly here because there are probably several men present who are not well versed in the language of the dyehouse.

As mordants for the Logwood we have the metallic salts of iron, of copper, and especially of chrome. These metallic salts are not only the so-called binders between the Logwood, or Hematin, and the fiber; they serve also as the oxidizing agent whenever it is necessary to transform the Hematoxylin, the lower oxidized part, into Hematin, or bring the Hematin out to its full development. It is therefore very important to establish between the given Logwood, the fiber, and the mordant a certain equilibrium, or let us call it "balance."

If we take a highly oxidized mordant and bring it together with a more highly oxidized Hematin, and let them work together, we can expect some trouble. The oxidizing mordant will not only develop all the Hematoxylin present and bring the full development of the Hematin before us, but it will go even farther; it will attack the Hematin and destroy it partly. We will get not only loss of color but a very disagreeably dyed material.

So I repeat, it is necessary to find a balance between certain grades of extracts, certain material and the necessary mordant.

After many years of observation, of trials in laboratories and in factories, we found this balance, that is, the right proportions between the Logwood, the fiber and the mordant.

It is my intention to bring before you certain methods (and only those methods of which we are sure that they give good results) in which the right balance between the extract and the mordant has been established. It may be that I won't give very many new points, but you will find in my talk suggestions that perhaps even the chemist working on synthetic dyes can take some notice of.

Of course, for each individual case these methods have to be altered somewhat to fit the given conditions.

Let us consider cotton first. This fiber was dyed extensively with Logwood till the direct blacks and sulphur blacks appeared on the market. The Logwood extract itself gave good results, but the methods for its use required more time and more handling of the material than the synthetic blacks. Thus it was up to the manufacturer of Logwood extract to meet the competition of the synthetic blacks by finding simpler and cheaper methods.

The method which we especially recommend to get somewhat near the cheapness of the sulphur black or the direct black, is a method that is based on the formation of a lake between bluestone (copper sulphate), soda ash, and Logwood. We use here the lower form of the Logwood extract, and the dye bath is easily made up, under certain precautions.

I suppose you are familiar with the machines that are used for the dyeing of raw cotton—the Giles machine, the Delahanty, the Klauder-Weldon, and perhaps others. When using these machines we dissolve a certain amount of soda ash in the boiling bath, put in the necessary amount of Logwood, and then add slowly the bluestone previously dissolved in hot water. It is necessary to do that very slowly, otherwise you will have a strong formation of carbonic acid and foaming. When this bath is ready the machine turns and immerses the cotton in the

Logwood bath; it will come out of the bath into the air, where oxidation takes place. By the action of the copper sulphate on carbonate of soda, carbonate of copper is formed, which itself is decomposed into copper oxide, an excellent mordant for the Hematoxylin. A good blue-black is the result. This black can be greatly improved by an after-treatment with chrome, and can be shaded with Fustic.

You will ask me, of course, "Why should I apply such a method to the dyeing of cotton when I can dye it perhaps cheaper and quicker with direct black or with sulphur black?"

This is the answer: Logwood possesses one quality which scarcely any other dyestuff has, namely, weight-giving and penetrating of the fiber. This weight-giving property is still the important factor in using Logwood on cotton. It is an established fact that cotton dyed in that way with Logwood will gain in weight up to 5 per cent and will produce a full-feeling fiber.

When dyeing cotton with sulphur black a certain loss of weight will occur and the fiber itself will be weakened somewhat if the washing of the cotton is not carried out carefully. To overcome these two points of defects some mills dye part of their raw cotton with sulphur black and another part with Logwood extract. By carding and spinning together the two differently dyed fibers a full feeling and strong thread is obtained.

There are also certain classes of cotton-piece goods dyed best with Logwood, as the latter will give better penetration and weight-adding than synthetic blacks. For goods like linings, book-binding cloth and generally all strongly woven cloth, which has to be dyed cheaply and in great quantities, Logwood can be profitably used.

For the dyeing of such goods we recommend the following method: The pieces are first impregnated with a Logwood solution, then dried, chromed and washed. The dyeing and chroming operations are carried out in small dyehouses on jiggers or padding machines. For mass production a better arrangement has met with good success. The cotton pieces sewed together, like a continuous band, enter a box machine, fitted out with rolls and two nips. The machine is fed by a pipe from a tank with a Logwood liquor containing a small amount of acetic acid. Leaving the box machine the pieces are led over a set of dry-cans, thoroughly dried and run immediately into a box fed from a tank by a pipe with chrome liquor. From the chrome bath the pieces run through a washing machine to a stentering frame and are ready for the finishing. The whole operation is a continuous process, the pieces running along at a speed so that from 70 to 100 yards per minute pass through.

The men who use this method claim that Logwood dyed lining cloth will stand hot pressing better than cloth dyed with other dyestuffs. Let us turn now to the use of Logwood on wool.

For the dyeing of wool the cheapest and so far the best mordant is a chrome mordant, and here we use the higher oxidized Logwood extract, the Hematin. Perhaps, later, in another talk, I can explain why we use the higher oxidized Logwood for wool. I think it might be well to give you a plain explanation about the working of the mordant for the benefit of those men present who are not so fully informed.

You know that when you boil wool in a solution of bichromate of potash or soda, the wool will decompose the bichromate, and in such a way that the decomposed product will become fixed on the wool partly in the form of chromic acid and partly in the form of chrome hydroxide. The wool will then have a yellow color. If you would take such wool, prepared with only bichromate of soda or bichromate of potash, having therefore big amounts of chromic acid, you would, as I explained before, work havoc on the Hematin, because you would surely over-develop it and destroy the valuable dyestuff. For that reason it was proposed to use along with the chrome certain agents which will decompose the chrome in such a way that the result of the chrome mordant on the fiber will be more of a chrome oxide or chrome hydroxide. This reduced mordant, together with Hematin, naturally will form a lake; perhaps a certain part of chromic acid, which might still be present, will work on the Hematoxylin, but only in a mild way, and we will have, therefore, in such a reduced mordant an excellent means of developing our Logwood on the fiber.

Such method has been worked out. The mordant is made by using 3 per cent of chrome, 3 per cent of half refined tartar, or 2 per cent of tartaric acid, and 1 per cent of bluestone.

When the bath is ready, we put the wool in it at a temperature of about 140 deg.; we bring it slowly up to boil, and keep it as near to the boil as possible for from one hour to one and a quarter hours. Then the wool must be washed very carefully. Naturally, if you leave a surplus of mordant there you can expect that the color lake which is later formed will not be fastened on the wool but will make the ultimate result smudgy.

After washing the wool very carefully, it is immersed in a bath containing Hematin paste, and along for shading certain yellow coloring matters. I will come to the yellow coloring matter later on. The wool remains in the dye bath for about 1 to 1¼ hour, when it is carefully washed and dried.

Now I will speak a little about this mordant, why it has been applied and why it is still in use. The reason is that you can apply on this mordant, which is in full harmony with Hematin, a large amount of Hematin, and there another good quality of the Logwood comes to the front. You can use big amounts of the color. The mordant is capable of taking up 2 per cent, 5 per cent, 10, 20 and even more. The resulting shade with 2 per cent will

be light blue, with 5 a medium blue, with 8 to 10 a blue black, and as you put more dyestuff on the black will become intensified.

I do not intend to "knock" the synthetic dyestuffs, but if you would try to intensify a black, using a synthetic black, by putting more and more dyestuff on a certain amount of chromed wool, the result would be disastrous, because there is a limit to the amount of synthetic black you can put on chromed wool. When the maximum is reached, no more will go on. Of course, you can say, "Well, I could put on more chrome," but it doesn't do the wool any good to put on more chrome. Three per cent, it has been found in practice, is just the right amount to be applied. If you apply more chrome on your wool, the wool will become harsh, and perhaps break in the spinning. I am speaking now about raw wool.

But this mordant will fasten big amounts of Hematin, which enables us to intensify the shade of black.

I spoke before about shading the black, which has always a bluish cast. The requirements of the trade change. This year a blue-black is necessary. It may be that in three months a jet black and a few months later a brown-black is looked for. The Logwood can take care of that. If a blue-black is required, there it is. If a jet black is required, we add some yellow dyestuff.

Among the yellow dyestuffs that have been used are Milling Yellow, Tartrazine and Chrysophenine But the old-timer, Fustic extract, is still used by most men who use Logwood, because these two bodies work best together. Besides, these synthetic dyestuffs are not always as fast as the claims that are made for them would lead us to believe. I have no intention of entering into a discussion about that at this time, because it would take a long time to explain these things, but the fact remains that more Fustic is used for shading Logwood than Tartrazine, Milling Yellow or Chrysophenine.

For browning or reddening Logwood, Alizarine Red can be used. As Alizarine is a dyestuff which can be easily fastened with chrome, you have here an easy procedure; you can use a mordant which not only fastens the Hematin, the Logwood, carefully on the wool, but also a mordant which is able to take any other dyestuff that may be necessary for shading.

The standard method can be applied to piece goods, raw stock, slubbing, in fact, to wool in any form. For example, woolen stockings with white cotton parts (toes and heels), can be dyed by this method. There was a time when it was said that Logwood could not be used for this purpose. It is now used to quite a large extent for this kind of goods. If you will apply this mordant, which we now call the Standard Mordant, on stockings (they must be really woolen stockings) with toes and heels of cotton, you will get a very fast dye. In order to clear the cotton, which will surely be a little bit stained in dyeing, you can apply a weak chlorate of lime bath. The chlorate of lime must not stand higher than half a degree Twaddell. By doing that, you clear the cotton completely, without attacking the wool. Besides, you submit the wool thereby to a process about which there was so much talk some years ago, namely, a process which makes unshrinkable wool.

It is not within the plane of my little talk to speak about unshrinkable wool, but you see how Logwood enables us to apply one process together with another one to get a product which the manufacturer could not otherwise get without applying a special process.

Besides the standard method (3-3-1, as we call it), we have, of course, older methods; for instance, the use of copperas (iron sulphate), bluestone (copper sulphate), oxalic acid as mordant, with the Hematin bath as coloring matter. It has even been suggested (and been carried out) to use all the ingredients in one bath, dissolving the bluestone, the copperas and the Logwood together, and dissolving the resulting lake in sufficient oxalic acid to keep it in solution. You will still find dyers using these methods, but the majority of dyers use our standard method.

It was explained before that between the mordanting and the dyeing of the wool there is always a careful washing. This careful washing naturally takes time. In our American life, time means overhead, which takes most of the profit out of a mill. Therefore, it was up to the chemist engaged not only in the manufacture of natural dyes but also in synthetic dyes, to find a method by which this washing could be eliminated and the process made shorter; in plain words, to find a one-bath method. We have worked—when I say "we," I refer to several chemists working in the natural dyestuff line—for years on the proposition.

We have succeeded in finding such a method, using formic acid as reducing agent for the bichromate, whereby the chrome-bath becomes free of chrome, so that the same bath can be used as Logwood dye bath.

I will explain this method a little bit further. Suppose you have a circulating machine full of wool, where the proportion of the wool to the water is often 1 to 6, whereas we usually dye wool 1 to 50. Men who are in the business will tell you that it is sometimes difficult to use these machines properly because the manufacturer wants plenty of wood dyed in the machines; they cost money. But when the machines are packed full of goods, there is very little room for the dye liquor. Therefore, you must have dyestuffs in this small amount of water which are fully soluble, so soluble that the packed wool on the lower part or upper part of the circulating machine will not serve as a sieve to keep back dyestuff which is not dissolved. Keep that in mind when carrying out the dyeing of Logwood on these machines, a body which

always appears to us as something that is not so very soluble.

First, we fill the machine with wool; we add water, and let in steam; the chrome, previously dissolved, is put in; the circulation is arranged; the formic acid is put in, in parts. The color of the liquor is observed; at first it is of a yellow cast; it becomes slowly clear, and nearly colorless. That proves that all the chrome is fastened on the wool. Without washing, without taking the wool out, we put right on top of the wool the predissolved Logwood; we let the circulation proceed, and we get a very fine wool, fine in shade as well as in feel.

When making a trial I took particular pains to note whether the wool on top was different from the wool in the middle or on the bottom. I could see no difference. But in order to carry out this method, it is necessary to have a specially well soluble Logwood. We have such a Logwood.

I mentioned before that in the trade Logwood is known as being oxidized to a certain percentage. We have succeeded in oxidizing Hematin, Logwood paste, to 96 per cent. Therefore, we have made a "C. P." Hematin. I mean by "C. P." commercially pure, not chemically pure. (Laughter.)

We have such Logwood. It is very soluble. When you put ordinary Logwood paste in cold water, it forms a very smeary brown mass. In medium warm water, it would dissolve. You need water of at least 160 deg. to keep your Logwood in somewhat good solution. This Logwood which we have now in paste form will dissolve in water of about 100 deg. Fahr.

In using such a process, you must have men who are somewhat trained. Unfortunately, there are not so many dyers who will use processes that require them to think a little bit, or which will require them to stand by the machine for a few weeks and do the dyeing themselves. It is much easier to follow a certain formula which has been used before, and let it go at that.

In France and England they have developed two other formulas based on the same principle; in plain words, a one-bath method. I hope I may, later on, have occasion to explain these methods also to you.

As we have made only a limited number of practical tests with the one-bath method we are not ready at the present time to judge about the merit of this method. When more practical tests are made later on we can do this better and bring the method more into the trade.

So much about this one-bath method. I pointed this out to you only to show that we chemists of the natural dyestuff industry have not been asleep.

I come now to the most valuable fiber, valuable from the point of view of the manufacturer as well as the consumer, namely, silk. I understand silk now costs about $7 a pound. Before explaining the method, I presume that many of you are not very well acquainted with the application of mordants on silk, so that I should like to go a little bit into the detail of that subject.

On the silk fiber, the good properties of Logwood are brought out more than on any other fiber. Logwood when applied to silk shows an unsurpassed property to give weight, to fill the fiber, besides producing a very deep, splendid blue-black. It might be considered that on cotton and even on wool the synthetic blacks have superseded the Logwood, pushed it out partly, but there is so far no black on the market that has any influence on the selling of Logwood as far as silk is concerned.

We know that the silk as it comes to us from India and China is not a very nice-looking material. It looks like horsehair. This material has to be boiled off with soap to bring it into form so that it can be used successfully in the other operations of the mill. But what happens when you boil off silk? You lose up to 25 per cent, in round numbers, of your weight.

If a silk manufacturer pays $7 a pound for silk and he loses one-quarter or 25 per cent on his raw material before it goes into operation, naturally he wants that 25 per cent added; not only that, but something more, if possible. (Laughter.)

It so happens that there are certain chemicals which work together with silk in such a way as to give incredible weight to the silk. It is not necessary to use too much of these chemicals. There are, of course, some men who overdo it. I have never seen it done, but it has been reported in the trade that silk has been weighted up or loaded, as it is called, to 300 per cent. (Laughter.)

I will go back now to the method. Boiled off silk when immersed in iron liquors, and especially if nitrate of iron is used, will take up the nitrate of iron. Of course, that adds a certain weight. When the treated silk is put in a tannin substance, like Divi Divi, Cutch, Gambier, Fustic, Quebracho, whatever is necessary or advisable to use in the special case the silk mordanted with iron will take up the tannin and naturally will form an insoluble iron tannate. This iron tannate, on the other side, has the property of taking up large amounts of Logwood.

Now then, when an honest man wants to make a black with Logwood, he can do it in a simple way. It is known in the trade that there is nothing dishonest about dyeing silk with Logwood. You have to use iron and a tannin substance. Well and good! But when you give a simple treatment with tannin, with iron and with Logwood, your silk which was previously boiled off to 12 ounces to the pound will be now between 14 and 15 ounces. There a man brings a product on the market which is absolutely honest. If he wants to, he can get his money back by putting a higher price on it. But most of them are not satisfied with that. (Laughter.)

You can very well understand that I can make a strong-

er iron liquor, or a stronger tannin substance, or add more Logwood, and I can repeat this process and pile on these unnecessary substances indefinitely. It is within your power to produce 12 ounces, 14, 16, 18, 20, 22 or 28 ounces. What does than 28 ounces mean? It means that that silk which was formerly 12 ounces to the pound has become 28 ounces. (Laughter.)

This process is carried out by immersing the silk in iron, washing, putting it in a tannin substance, rinsing, exposing to the air, putting in the Logwood, rinsing and drying. But there again comes the question: Is it not possible to dye silk without all these apparently unnecessary washings? There are such methods, and they are used successfully on piece goods, especially in England, on goods that must not be handled very much—pile goods, such as velvets, plushes, and so on.

This method avoids unnecessary washing. It is done in this way: You dissolve, preferably, Fustic (because it serves at the same time for shading), bluestone, copperas together in one bath. You get different shades, according to the temperature applied whether it is 160 deg., 180 deg., or 190 deg. Fahr

You dye your silk piece goods either on winches (I presume you all know what a winch machine is), on jigs, or on the more modern silk dyeing machines, for about one hour. A slight rinse is necessary usually, but not always.

You have done away there with one process, that of immersing your silk first in iron, washing, and then immersing in tannin. You combine the different operations and then dye with the Logwood. Good results have been obtained by this method. It is used, as I mentioned before, on velvets, on fine satins, and so on.

Another and more modern way for preparing silk so that it will take up Logwood quite readily is the so-called tin phosphate process. Much has been published about this process, thus I presume that you are somewhat familiar with it. Nevertheless, a short resumé might refresh your memory.

The procedure is this: You immerse the silk in a bath of bichloride of tin which stands about 50 deg. Tw. or 30 deg. Be.; the bath bichloride of tin is kept as cold as possible. After one hour, you take out the silk, give it a cold wash, a medium wash and a wash that comes near to 140 deg. Then you immerse the silk in a bath of phosphate of soda at 149 deg. You leave the silk in a half hour, then wash backwards, first warm, medium warm, then cold.

Such a procedure is called 1-pass silk. If you repeat that it is 2-pass; another one is 3-pass; some use 4-pass.

The silk takes up by 1-pass 2 ounces of weight and becomes 14 ounces; by 2-pass it takes up 5 ounces and becomes 17-ounce silk; by 3-pass it takes up 8 ounces and becomes 20-ounce silk. One-pass silk, originally 12 ounces, will be 14 ounces; dyed with Logwood, one simple bath, with about 80 per cent Logwood in the dye bath, it will become 17-ounce silk. You can make with one pass of tin phosphate silk, and a Logwood bath, without overdoing it, a silk which is 17 ounces. That means it is even over par. If you take a 2-pass silk, whereby the silk becomes 17 ounces by the tin process alone, and give it a Logwood bath, the silk will become nearly 24 ounces. That means it is possible to make 2 pounds of silk out of 1. Three-pass silk will give even a higher weight, 26 ounces, and perhaps more.

If the manufacturer keeps down to 2 or 3 or 4 passes, there is no danger; such silk, when dyed properly and carried to the trade in such shape that it is used up after a certain time, will pass.

I observed that when the silk was given 4 passes, instead of acquiring more weight, it lost weight, and with 5 passes I got scarcely any good results; the silk was no longer silk; there was only a skeleton of tin phosphate there (laughter), which was kept together by the help of Logwood and such fibers of tin as were there.

I don't wish to speak too much about the dyeing of silk with Logwood because that is such an enormous theme that it ought to be discussed by men who are engaged in that trade; they ought to come before you and give you their practical experience. I have done some interesting work on it, but not enough to give you all the interesting facts which it is necessary to know, and I hope that your Chairman will arrange a meeting for some future time when men who are better acquainted with the silk trade can give you more information along this line.

The same remark applies to leather, which is dyed extensively with Logwood. The leather men of your association will surely give you a better account of the use of Logwood than I can give you.

I would like to talk to you now about one silk that has come into great use, and I have a few samples of it here which I will describe later on. The material I refer to is tussah silk. It comes, like real silk, from India and China. Tussah is a product from a silkworm, but a tougher silkworm than the one producing real silk. The toughness or stiffness of the fiber gives it an enormous advantage. Here again Logwood is a big factor to help this article along. Tussah silk is much stiffer than real silk and therefore is more suitable for the manufacture of long-pile fabrics, that is, plushes. As many of our ladies like to be clad in splendid looking garments, the silk manufacturers have answered the call by making a fabric out of this tussah silk which has all the appearance of sealskin.

Here [showing sample] is a plush. The warp is sulphur dyed. On top is the long stapled tussah silk. It looks very rough, and hasn't a very nice appearance. Here you have the same material partly finished. What has happened? The pieces of silk were first dyed with

a tannin substance, and for certain reasons, Gambier and Cutch applied. After they had been treated with Gambier and Cutch, they were chromed, the tannin matter and the chrome filling up the fiber somewhat, as you see here, giving it a brown bottom. The Logwood is later put on this brown bottom by dyeing. So you have there the so-called sealskin plush.

When you blow real sealskin, the fur always shows brown on the bottom, and only black on top. Some manufacturers have even asked for a method by which they could leave this bottom brown and put the black only on top.

Logwood has also answered the call. Here you have such an imitation of sealskin. The material was first treated with Gambier-Cutch, and then chromed. The Logwood was brushed on the top of the material with the help of the so-called tipping machine. By steaming the material, the Logwood fastened itself on the surface of the fiber.

Logwood fills out the fibers, so that when these goods are finished you have a material that, if you are not very well acquainted with real sealskin, you can give to your sweetheart or wife and say that it is sealskin. (Laughter.)

The same method as applied for tussah silk has been made use of recently on a quite different material. I show you here a plush, the face of it made of rayon, the back—the warp—a mixture of cotton and wool. The manufacturer of this product found that he had difficulties not only with the dyeing of the three different fibers, but also with the empty feel of the dyed article. The method using Cutch-Gambier, Chrome, Logwood solved his problem, producing a good level shade and fullness of feel.

Therefore, Logwood can be used on materials where other dyestuffs simply fall down. Wood, for instance, is one that comes to my mind just now. You all are familiar with the little black piano keys. If those keys were made, as they were formerly, out of ebony wood, you would have to pay a pretty steep price for a piano. Most of this wood is dyed nowadays with Logwood by a very interesting process.

The piano keys are placed in a big kier. This kier is connected with a vacuum pump and all the air is drawn out. When as complete a vacuum as possible has been acquired, Logwood liquor is pumped in, plain Logwood, with a little bit of soda ash, and air pressure is applied. In other words, on the one side all the air is taken out of these keys, whereas Logwood as much as possible is pressed in.

When the keys have been in the kier for about three hours, the liquor is let out to be used again and pyrolignite of iron put in, again under pressure. Then these keys are taken out and washed, and they can be polished wonderfully. You can open keys dyed by this process and you will see that the penetration is good. I don't know whether synthetic blacks will do that. I have no intention of knocking any synthetic black, and I have no right to do that. Logwood is not perfect; it has many disadvantages. For instance, you will tell me right away that it does not stand acid treatment. Granted. But it will stand acetic acid, and it will stand all other requirements which synthetic blacks will stand. It is not a cheap black. Therefore, it is used on high-grade material. It is not a black which is dyed very quickly. It takes time for dyeing. But see what a product you get! You get a product which is a standard. Wherever blacks are compared, you hear as a rule the statement, "It comes up to the Logwood shade," and I think that is the biggest compliment that can be paid to any article.

Sometimes it seems to me that Logwood is in about the same position as my favorite humorist, Mark Twain, was in. He was told that he was supposed to be dead, and was even shown some papers which printed the story that he was dead, so he sent the following telegram to one of the editors: "I would like to inform you that the news of my death is grossly exaggerated." (Laughter.) Wherever a new black is invented, it is said, "Now Logwood is dead." We know that Logwood is not used so very much now, but it isn't dead, and when we have this one-bath process completed I think we will again be in a position to successfully carry on our competition against synthetic blacks.

Gentlemen, I thank you. (Applause.)

Discussion

Chairman Grandage—I am sure we are indebted to Dr. Lesser for this most interesting discourse on Logwood black. Dr. Lesser will be quite willing, I am sure, to answer any questions that any of our members may want to ask.

Dr. Chapin—I would like to ask Dr. Lesser his opinion concerning the effect of proper weighting of silk.

At a meeting about six months ago the question was raised as to whether the weighting of silk did not tender the fiber. What is your opinion about the effect upon the strength of the silk fiber and its resistance to wear and tear when it is properly weighted?

Dr. Lesser—I have some pieces and some yarn in my place which were dyed very carefully by that process, which are at least four years old, and I must say they are just as strong now as they were before. How long they will remain that way, I do not know, but I think it depends a great deal on the way in which these pieces are packed, and the way they are stored. That has a great deal to do with it. When you place pieces of silk on a shelf, naturally, the silk is in folds and one piece is placed right on top of another, and there is a certain pressure. I think even silk which was not treated with tin would become somewhat tender at those places.

Secondly, the temperature of the room in which the

material is kept, and the moisture will have an influence on it.

That is a very interesting point and I will make some trials along that line. If I am not mistaken, I have already made such trials, where I exposed silk dyed black to moisture, for a long time. When I find that test, I will inform you.

Of course, this point enters into it: If a silk has been tinned and the ultimate treatment has not been completed there is a danger that some of the salt may be left in the goods. It has even been found that such goods have become red. As far as I can recall, trials have been made by Professor Hermann, the man who was really the originator of this method. He has proven that common salt left in the goods will make it tender and produce red spots. That is as far as my experience goes. But how long that silk will stand, I cannot answer definitely. I shall look up my notes on that.

Mr. Hayes—You referred to dyeing of cotton with Logwood black by running through the Logwood, then chroming, steaming, and washing. I would like to ask: In a process like that, what factors influence the fastness of that Logwood to rubbing?

Dr. Lesser—The most important part of that procedure is that when these goods have run through the Logwood machine and run over the dry cans, they must be perfectly dry when they leave the dry cans. If the goods are not absolutely dry, you will get spots or an ultimate rubbing will take place there. I have seen pieces which were run through such machines; the man wanted to save steam and didn't dry the goods sufficiently. The result was that there was rubbing. Then, of course, good washing is essential. An overchroming will also produce rubbing.

Mr. Hayes—You don't mean overchroming, you mean underchroming, don't you?

Dr. Lesser—Overchroming is more dangerous. I think I can give you the figures on that from one example I know of. In that box there were 80 pounds of Logwood extract and 5 gallons of acetic acid. That was the liquor used for dyeing. There were 250 gallons of chrome liquor, 49 pounds (I remember that exactly) of bichromate of potash, and 5 pounds of soda ash.

Mr. Hayes—At what temperature did you run the chrome bath?

Dr. Lesser—The chrome bath was kept at 140 deg. That is high enough, because if you have it too high (we tried it) you are liable to strip off too much of the Logwood and get your chrome bath too dirty.

You can arrange, if you haven't such a machine, to dry it on jigs, bring it on dry cans, or put it somewhere for drying, then put it on another machine, chrome it, and put it somewhere else for washing. I have done that, too, in factories where they did not have the continuous machine. You can do that and get an excellent black, cheaply, too; it doesn't cost very much.

Mr. Hayes—You think the biggest factor then would be the complete drying of the goods before chroming?

Dr. Lesser—Yes, and you must wash the goods carefully. The washing machine must be arranged so that the wash water must run out when dirty, not as it does in most places—at the end of the machine where the clean water comes in; arrange your machine so that the dirty water runs out at the end where the dirty goods comes in. In addition to that, have a sprinkler there, so that as much water as possible can be sprinkled on.

Mr. Jackson—I would like to ask you, with regard to that Logwood tin weighting method, whether you have ever tried to use iron sulphate instead of phosphate and what would the effect be?

Dr. Lesser—I have never tried it, but I think iron has been used on top of tin, and the results were not very satisfactory. When that is done, I have been told (I have not tried it myself), the silk becomes tender; that is, when iron is used on top of the once tin-phosphated silk. Is that what you mean: using iron on top of the tinned liquor itself?

Mr. Jackson—Yes.

Dr. Lesser—No, I have never used it, but I should imagine that this method could be used only for black. The tin phosphate process is used because the silk is kept colorless, or let us say, white.

E. F. L. Lotte—Iron and sulphate do not work very well with phosphate. You could use tin and sal soda, or sodium carbonate.

Dr. Lesser—Right!

Mr. Lotte—Then you could use nitrate of iron or iron sulphate. But you do not get the weight by that method, and it is not as economical. That is an old method that was discarded years ago. When they originally used tin, they neutralized the tin with sodium carbonate, and then they gave the red iron, as we call it, or nitrate or iron, or the iron sulphate, if they used that. But there is apparently some action when phosphate and iron sulphate are used that is not explained; the fiber is not left in as good condition as it should be.

There were some experiments made on that during the war, in order to save Methylene. In order to avoid the necessity of giving Methylene Green, or Blue, and Chrysoidine, they used iron sulphate. We have not been able to explain in our laboratories why that does not work.

Probably that answers your question.

Mr. Jackson—Yes.

Mr. Lotte—There are some things I would like to ask the doctor. He spoke about blacks that lasted four years. Is that correct?

Dr. Lesser—Yes; I have some.

Mr. Lotte—I believe you are correct. I won't dispute

that. But I would like to ask, then, why are they dishonest?

Dr. Lesser—Dishonest? (Laughter.) I would not say that. Perhaps I was a little bit too emphatic in using that expression, and I will take back that statement. But they might be dishonest very soon. As far as I know, it will be something like the food law; we have a Pure Food Law—

Mr. Lotte—I don't think they are any more dishonest than eight or ten karat gold.

Dr. Lesser—All right, I stand corrected. I take that back. (Laughter.)

Mr. Lotte—There are just a few more things I want to say.

You spoke, Doctor, about a black that was given just one iron, then Logwood, and then put out. I feel that we have had more dishonesty in that black than in any other black that has ever been put out in the silk business, and I will explain it in this way: That is supposed to be a fast black. For a tailoring dye, for years the test was the seaman test of oxalic acid, to find out whether it was fast or whether it was extra weighted, or not. That black was supposed to be a fast black, and you know that Logwood does not hold unless it has something there to hold it.

Dr. Lesser—Exactly.

Mr. Lotte—It had very little to hold it, and it was the most fugitive black ever put on the market. We put it on because the trade demanded it, but I felt it was not honest because it did not hold the color. Not even the synthetic blacks are as fugitive as that black. When we get into the heavier weighted black, we have a black that stands.

When the Government wanted a black for her neckerchiefs, you will find that she required 4 per cent ash, which means quite a good bit of red iron, and she had that on there in order that it might hold the Logwood. If a smaller amount of iron was used in what was called a "fast black," because it was pure, it would not stand the Government test for her Navy neckerchiefs, because the salt water stripped the Logwood. So that in some cases, in fact, in a great many, Logwood requires a mordant, as we call it, or something, in order to retain the Logwood, to make it fast.

Take blacks which are given prussiate, which you said was put on in order to pile more iron on, and I might say that those prussiate blacks are fast to light, and fast to soap. I think you will agree with me on that.

Mr. Lesser—Certainly.

Mr. Lotte—And if you will follow them, you will find that in a good many cases the trade has been benefited by those very things, and your Logwood has been made to stand simply because we did weight some of the silk up a little higher than we would if we had given a pure dye black.

Dr. Lesser—Thank you for the information.

Stewart F. Carter—I would like to ask Dr. Lesser a question in regard to Logwood liquor and Logwood extract, which has been a very mooted subject for some time back.

Take, for instance, a Logwood liquor at 12 deg. Tw., and a Logwood extract at 48 deg. Tw.

It was claimed that the 12 deg. Tw. liquor was proportionately stronger than the 48 deg. Tw. extract.

That is, if you took the 48 deg. Tw. extract and reduced it to 12 deg. Tw., this liquor would not be as strong tinctorially as the liquor which had been simply carried to a concentration of 12 degrees.

The idea was that in carrying the concentration up to 48 degrees there was some loss of coloring matter.

Did you ever come across anything like this?

Dr. Lesser—No.

Mr. Carter—That is a matter which has been often tried and my personal observation has been that such seems to be the case.

Chairman Grandage—Are there any further questions that you would like to ask Dr. Lesser? If not, the hour is somewhat later than the hour of adjournment at last month's meeting, which I had so much trouble breaking up.

If there are no further questions, are there any matters that any of the members want to bring before the Section at this time?

D. P. Knowland—I move we extend a rising vote of thanks to Dr. Lesser.

. . . The motion was seconded and unanimously carried. The meeting adjourned at 10.15 p. m.

Applicants for Membership

Active Membership

McKee, John L., salesman and demonstrator, John Campbell & Co., Charlotte, N. C. Sponsors: Malcolm Mackenzie and N. R. Vierira.

Murphy, William T, overseer of dyeing, Pacific Mills, Lawrence, Mass. Sponsors: Wm. H. Cady and George A. Moran.

Ordway, Charles B., dyer and textile chemist, Spalding Knitting Mills, Griffin, Ga. Sponsors: B. R. Dabbs and E. P. Davidson.

Shendel, Jr., John N., dyer, S. Roseman & Co., Philadelphia, Pa. Sponsors: J. H. Hennessey and B. K. Archer.

Van Mater, Henry L., chemist, American Dyewood Company, New York City, N. Y. Sponsors: D. P. Knowland and A. Elssmann.

Junior Membership

Glidden, Harlan E., student, Philadelphia Textile School, Philadelphia, Pa. Sponsors: E. C. Bertolet and P. Theel.

Graves, Albro N., textile chemist and colorist, Franklin Process Company, Providence, R. I. Sponsors: A. N. Dana and W. H. Wingate.

Changes of Address

Alter, Frederick A., 227 Ash Street, Willimantic, Conn.

Biach, Dr. L. K., consulting chemist, Ramapo Finishing Corporation, Sloatsburg, N. Y.

Callahan, Charles A., 576 Gage Avenue, South Memphis, Tenn.

Costello, John L., 3323 West Penn Street, Philadelphia, Pa.

Dalton, J. H., 107 Main Street, Andover, Mass.

Dengler, C. E., 134 Buckingham Road, Montclair, N. J.

Guiterman, E. W., 66 Marlboro Street, Newton, Mass.

Houghton, Robert, 14 Lincoln Street, Stoneham, Mass.

Jacoby, Morris, 384 East Twenty-seventh Street, Paterson, N. J.

Moorhouse, Wm. R., care of National Aniline & Chemical Company, 40 Rector Street, New York City.

Morrill, Elliott N., 1229 E. Knox Street, Galesburg, Ill.

Peacock, William H., 151 El Mora Avenue, Elizabeth, N. J.

Pietzsch, Otto T., 39 High Street, Passaic, N. J.

Raskin, Nathan, Banner Silk Dyeing Corporation, 214 E. Twenty-second Street, New York City.

Roos, Louis, J., 8 Thomas Street, New York City.

Runnells, Harold N., Box 138, West Sand Lake, N. Y.

Steele, E. V., 649 Westford Street, Lowell, Mass.

Stone, Charles H., 1101 Queens Road, Charlotte, N. C.

Sturtevant, F. W., 60 Grove Street, Lowell, Mass.

Tanguay, Gerard, 118 Mt. Washington Street, Lowell, Mass.

Taylor, Oscar C., 10 Stoughton Avenue, Webster, Mass.

Thompson, Jr., A. R., 90 Realty Building, Charlotte, N. C.

Williams, Charles, 123 Oklahoma Street, Springfield, Mass.

Worthem, C. T., 2315 Genesee Street, Utica, N. Y.

The Fifth Annual Meeting

of the

American Association of Textile Chemists and Colorists

will be held in Boston, with sessions *Friday evening, December 4th* and all day *Saturday, December 5th*

The Council is considering holding an *Open Forum*, Friday evening, where opportunity will be given to discuss subjects of interest to members.

If you have in mind any subjects which you should care to have discussed at this time, or if you are willing to open or take part in the discussion of any subject, kindly advise either the President or the Secretary immediately so that it may be printed on the final program, which will be issued in a few days.

ACETATE SILK—ITS DYES AND THEIR APPLICATION

(*Continued from page 752*)

injured. Possibly this process for mercerizing the cotton in Celanese cotton unions is covered by British Patent No. 210,484 to Wm. Marshall, dated October 5, 1922.

In this patent the inventor claims that the cotton yarns in mixed fabrics containing acetate silk and unmercerized or incompletely mercerized cotton may be mercerized by treatment with a caustic soda solution of 1.24 to 1.30 specific gravity at a temperature below 15 deg. Cent. (59 deg. Fahr.). The fabric should then be squeezed between rollers, stretched, washed, treated with cold dilute acid, and washed until neutral. It is claimed that with this process the acetate silk does not lose its luster or shrink, that the loss in weight is very slight and its dyeing properties are not materially affected.

Acetate silk is now being manufactured in France by the Societe pour la Fabrication de la Soie "Rhodiaseta" at St. Fons, Rhone, and in Switzerland As yet very little has been written regarding these products either in regard to their textile or dyeing properties.

Bleaching Acetate Silk

Besides the methods of bleaching given in Part II, page 238, acetate silk may be bleached in a hydrogen peroxide bath in somewhat the same manner as wool, except that the peroxide concentration should be somewhat higher than that used for wool. The temperature of the bath should not be above 100 deg. Fahr. (38 deg. Cent.) and only sufficient ammonia or sodium silicate should be added to render the bath very faintly alkaline.

The permanganate method of bleaching is also applicable to acetate silk. The bleach bath should be prepared with 1 pound of sodium or potassium permanganate per 100 gallons of hot water. The scoured acetate silk is worked cold in this bath for 2 to 2½ hours. As this bath becomes alkaline to litmus paper in use, acetic or sulphuric acid should be added to keep it very slightly on the acid side while the acetate silk is present. Usually about 100 to 200 c.c. of concentrated sulphuric acid will be required for each pound of permanganate used.

After the permanganate bleach, the brown manganese dioxide stains may be removed from the fiber by working them for from 30 to 45 minutes in a cold bath containing:

4 pounds of sodium bisulphite, and up to
2 pounds of 66 deg. Be. sulphuric acid in
100 gallons of water.

If the acid is omitted, the clearing bath may be warmed somewhat to accelerate the reaction, or the amount of bisulphite may be doubled. When the skeins are white, they should be rinsed with fresh water until odorless.

Bluing or Tinting Acetate Silk White

In case the acetate silk is to be used undyed and it is desired to improve the natural color without an actual bleaching operation, the fiber may be blued by tinting it in a bath containing a violet or blue dye of a reddish shade. The dyes most frequently used for this purpose are the Methyl Violets and Indanthrene Violets or Blues, although certain other dyes could be used for the purpose. In case the Indanthrene dyes are used, the prepared thick dye paste is simply diluted with water and added directly to the "bluing" bath without reduction.

(*To be continued in an early issue*)

UNEVEN BLUING

Sometimes it occurs that one or two pieces of white goods will come out of the wheel very blue, while the remainder of the load is evenly blued. The immediate cause is evident—that one or two pieces have absorbed an abnormal proportion of the dye used.

Two factors effect the above condition. The first is that some fabrics exhibit an extraordinary affinity for dyes and an unusual ability to absorb them.

There is, however, another cause. Frequently a load will contain one or more pieces which have had very harsh treatment at some previous time with strong alkali or bleach and which contain a considerable amount of oxycellulose, the result of the harsh treatment. This treatment, for instance, might consist in ironing a fabric from which the alkali has not been rinsed. Oxycellulose has a great affinity for color, much more so that natural cellulose, and, for that reason, more readily absorbs the dye used in the blue. One test for oxycellulose is that of applying a solution of Methylene Blue.

Tests may be carried out in substantiation of this explanation. We have repeatedly produced the identical conditions mentioned by placing a piece of goods known to contain considerable oxycellulose in a load with unharmed goods and have driven the bulk of the blue into the test piece. The laundryowner naturally has no control over the constitution of the fabrics he puts into the wheel except to make sure that his process is such that he is not producing conditions in the goods which will react detrimentally later. This, of course, will not keep out the transient piece which causes trouble. However, if care is taken to use no harmful alkali, and to positively control the use of bleach, the laundryowner is taking the greatest precaution to do away with this sort of trouble.

GERMAN DYE WORKERS THREATEN STRIKE

Cable dispatches recently intimated that there was the possibility of a strike among the workers of the German dye plants in the district of Frankfort-on-Main. This was the result of the refusal of the manufacturers to grant them the wage advances looked for.

The Use of Dyestuffs in Analytical Chemistry*

Use of Methylene Blue Hydrochloride Solution—Leuco Methylene Blue—Successful Uses of Methylene Blue for Quantitative Analysis

By FREDERICK WILLIAM ATACK

THE dyestuff of highest tinctorial power which is most readily obtained commercially of a purity sufficient for use as an analytical reagent is Methylene Blue (hydrochloride, zinc-free).

Methylene Blue hydrochloride solution first used by Knecht[1] for the detection of titanium, the method being capable of detecting less than 0.01 mg. of titanium (in the trivalent condition). The writer found it possible to detect very readily indeed less than 0.002 mg. of molybdenum (in the trivalent condition) by means of Methylene Blue hydrochloride solution[2]. For comparison it may be noted that Komarowsky (Chem.-Zeit., 1913, 37, 957) has found the method of detection by the formation of ammonium permolybdate sensitive to 0.006 mg. of molybdenum.

Before a new reagent such as standard Methylene Blue hydrochloride solution attains general use in laboratories, it is essential that a large number of applications should be available involving its use, and for this reason the writer considered it essential to make a careful survey of all possible uses of this reagent.

The use of Methylene Blue hydrochloride solution in analytical chemistry depends on:

(a) Its property of forming insoluble salts of definite composition[3], e. g., the perchlorate, the formation of which was found by the writer to be available for standardizing Methylene Blue hydrochloride solution, thus avoiding the use of titanous chloride[4].

(b) Its property of forming additive compounds of uncertain composition with iodine (Pelet-Jolivet and Garuti, Bull. Soc. Vaud. d. Sc. Nat., 1907, 30; Pelet-Jolivet and Siegrist, Bull. Soc. chim., 1909, (iv.), 5, 626). This led to the use of Methylene Blue by Sinnatt[5] for replacing starch in iodine titrations. An interesting application of this reaction by the writer may be mentioned; traces of acid may be detected in highly-colored solutions by using Methylene Blue to identify the iodide liberated on the addition of a mixture of iodide and iodate (actually all commercial samples of iodide examined were found to contain sufficient iodate for this purpose).

(c) Its reduction by certain reducing agents (e. g., stannous molybdenous, vanadous, titanous, etc., salts) to (colorless) "leuco" Methylene Blue hydrochloride solution. A most important point in this connection is that ferrous chloride does not reduce Methylene Blue hydrochloride solution, and hence many steels, and iron alloys generally, may be analyzed rapidly by this method without the necessity of first effecting the troublesome separation of iron.

(d) Oxidation of "leuco" Methylene Blue hydrochloride solution by means of oxidizing agents (e. g., chromate, vanadate ferric salts, nitroso-compounds, etc.[6].) This really formed the basis of the method used by Knecht and Hibbert[7] for titrating quinone and ferric sales with standard titanous chloride, in which Methylene Blue hydrochloride solution is added as an indicator; any ferric salt or quinone remaining maintains the dyestuff in the oxidized (colored) form, and hence the disappearance of the blue color indicates the completion of the reduction.

(e) Reduction of 1.2-diketones, oximes, etc., by standard titanous chloride, and determination of excess of titanous chloride by means of standard Methylene Blue hydrochloride solution. This avoids the necessity of titrating the excess of titanous chloride with a ferric salt, which frequently proves impossible when dealing with organic substances.

Methods for using standard titanous chloride are detailed in Knecht and Hibbert "New Reduction Methods of Volumetric Analysis" (second edition, Longmans-Green); a useful summary will be found in The Chemist's Year Book (Westman Press).

Methylene Blue hydrochloride may be considered simply as (M.B.)Cl. For example, precipitation with perchlorate gives: (M.B.) Cl O_4, and with dichromate: $(M.B.)_2$ Cr_2O_7. Reduction of Methylene Blue hydrochloride proceeds as follows:

$$(M.B.)\ Cl + 2H = (M.B.)\ H + HCl.$$

Leuco Methylene Blue

The use of leuco Methylene Blue hydrochloride solution for estimating oxidizing agents has certain limitations. The leuco Methylene Blue hydrochloride solution is not sufficiently susceptible to molecular oxygen to enable it to be used directly for estimating dissolved oxygen in water and in alcohol, but this was found to be possible using nitrate oxides as carrier (e. g., by adding a small amount of sodium nitrite and decolorizing again with titanous chloride). It was not found possible to estimate hydrogen peroxide or the

*Paper presented at Dominion Convention of Chemists, Guelph, Ont., June 4, 1925, and published in Canadian Chemistry and Metallurgy.

so-called (but not true) "per-salts" (e. g., commercial sodium "perborate") containing hydrogen peroxide of crystallization (see, for example, Riesenfeld and Mau, Ber., 1911, *44*, 3589).

Nitrates oxidized leuco Methylene Blue hydrochloride solution very slowly under the conditions used for titration purposes, and hence it did not prove possible to estimate commercial nitrites, which always contain nitrate. The difference between the usually accepted constitution of nitrites and nitrates may be noted in this connection. It was, however, found possible to estimate the nitrite content of pure silver nitrite[2] after conversion into sodium nitrite by addition to leuco Methylene Blue; a method for volumetric analysis of potassium by estimating the nitrite in the cobaltinitrite precipitate proved successful[2].

Another remarkable difference is in the behavior of chlorates and of perchlorates toward leuco Methylene Blue hydrochloride solution, the former effecting a rapid and quantitative oxidation, whereas in the latter case the reaction is too slow to be of any practical value.

Although small amounts of chlorine present in "synthetic" hydrochloric acid could be estimated by its oxidizing power on leuco Methylene Blue hydrochloride solution, nascent chlorine exerts a destructive action and hence hypochlorites cannot be estimated by this method. It follows that the reduction of chlorates by leuco Methylene Blue hydrochloride solution cannot therefore proceed via the interaction of chlorate and hydrochloric acid to give chlorine. In connection with the use of Methylene Blue for volumetric anlysis it may be remarked that the high tinctorial power of Methylene Blue enables N/500 solutions of the hydrochloride to be used readily for titration purposes provided suitable precautions are taken regarding the dissolved oxygen present (especially when carrying out titrations in alcoholic solution).

The advantages of the use of standard Methylene Blue hydrochloride solutions alone or in conjunction with titanous chloride have been fully summarized[2] and there is no doubt that its application may be even more widely extended, especially in view of the increased reliability arising out of its direct standardization against potassium chlorate[2]—a substance which can be readily obtained of a very high degree of purity, but previously not used as a standard—instead of the very indirect method (against ferrous ammonium sulphate via titanous chloride) introduced by Knecht (J. Soc. Dyers and Col., 1905, *21*, 9). In this connection it may be mentioned that the writer had noted in 1912 the decomposition of ferric thiocyanate (present in Knecht's method) which was later published by Philip and Bramley (J. C. S. Trans., 1913, *103*, 795); the equation for the decomposition given by these authors is incorrect, as they take no account of air oxidation, whereas the writer found that decomposition does not occur in carbon dioxide atmosphere. Knecht's method for estimating iron or for standardizing titanous chloride against a ferric salt using thiocyanate as indicator is therefore only accurate if carried out in an atmosphere of carbon dioxide.

The necessary Methylene Blue hydrochloride is a commercial product, and it may be mentioned that the standard (N/40) solution necessary can be prepared at less than half the cost of decinormal permanganate solution.

Successful Uses of Methylene Blue for Quantitative Determinations

Standard Methylene Blue hydrochloride solution has been used with success for the following quantitative determinations:

1. Hydrosulphites[7]; see also [4].

2. Titanium[8] by oxidation of the titanous salt ($TiCl_3$) to the tetravalent condition ($TiCl_4$) by means of standard Methylene Blue hydrochloride solution. Neumann and Murphy (Z. angew. Chem., 1913, *26*, 613) have found that this affords the only possible method for the direct estimation of titanium in presence of iron, silica and alumina.

3. Molybdenum in presence of iron (1) by oxidation of the molybdenous salt ($MoCl_3$) to the pentavalent condition ($MoCl_5$) by means of standard Methylene Blue hydrochloride solution.

4. Stannous choride and tin in presence of antimony[3], by oxidation of stannous to stannic chloride by means of standard Methylene Blue hydrochloride solution. (This provides another method for standardizing Methylene Blue hydrochloride solution in case titanous chloride is not available).

5. Chlorate[3], by reduction to chloride by means of leuco Methylene Blue hydrochloride solution, and estimation of the Methylene Blue hydrochloride formed (by titration with titanous chloride).

6. Chromium[5], after oxidation (by means of sodium peroxide) to chromate which is reduced to the chromic salt by means of leuco Methylene Blue hydrochloride solution.

7. Iron[6], as ferric salt by reduction to ferrous salt by means of leuco Methylene Blue hydrochloride solution.

8. Vanadium[5], after oxidation (by means of sodium peroxide) to vanadate which is reduced to the vanadic salt by means of leuco Methylene Blue hydrochloride solution.

9. Chromium, vanadium and iron in presence of one another by taking advantage of the fact that chromate and vanadium are reduced by boiling with hydrochloric acid, whereas only chromate is reduced by sulphur dioxide and neither of these reagents affects the state of oxidation of the ferric salt.

10. Perchlorate, in presence of chlorate[2] by precipitation of Methylene Blue perchlorate. This method has been used more recently by Monnier (Arch. Sci. phys. nat., 1916, (iv), *42*, 210) for the estimation of perchlorates in Chile saltpeter; this author does not

appear to have been aware of the writer's prior publication.

11. Potassium[2], by precipitation as potassium sodium cobaltinitrite, $K_2NaCo(NO_2)_6.H_2O$, which is decomposed by caustic soda, and the sodium nitrite in the filtrate determined by addition to leuco Methylene Blue hydrochloride solution.

12. Dissolved oxygen in water and alcohol[2], by addition to leuco Methylene Blue hydrochloride solution containing nitric oxide as carrier.

13. Nitroso compounds[2], by reduction to the amino compound by means of leuco Methylene Blue hydrochloride solution.

14. Quinones soluble in alcohol[2], by reduction to hydroquinones by means of leuco Methylene Blue hydrochloride solution.

15. Azo compounds[2], by reduction to hydrazo compounds, or further reduction in certain cases, by means of leuco Methylene Blue hydrochloride solution.

16. 1.2-Diketones, such as benzil[2], by reduction in alcoholic solution to benzoin by an excess of standard titanous chloride, and determination of the excess of titanous chloride by means of standard Methylene Blue hydrochloride solution.

17. Oximes, such as β-benzildioxime[2] ([10]).

It may be remarked that no known standard solution in common use as a volumetric reagent, for example, permanganate, can claim such a variety of possible applications; including particularly methods for the quantitative determination of organic compounds which could not be carried out by means of the well-known reagents. The general introduction of Methylene Blue hydrochloride as a reagent in analytical laboratories naturally depends largely on the extended use of titanous chloride, although the writer has found it possible to avoid the use of the latter reagent by using potassium perchlorate for standardizing the Methylene Blue hydrochloride solution[3].

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PERMUTIT CASE DISMISSED

The Federal Trade Commission recently dismissed the complaint, filed September, 1924, against the Permutit Company, manufacturers of zeolite water softening equipment, involving alleged unfair trade practices. After an exceedingly exhaustive investigation of this firm's trade practices, in which nearly a hundred witnesses were examined, including salesmen, officers and attorneys of a number of concerns engaged in selling zeolite softeners in competition with the defendant, the Commission finally arrived at an unqualified dismissal of its complaint. This decision is regarded throughout the trade as vindication of the company and a complete refutation of the charges of unfair practices against it.

Following is the text of the decision:

At a regular session of the Federal Trade Commission, held at its office in the City of Washington, D. C., on the 20th day of October, A. D. 1925.

PRESENT:

Vernon W. Van Fleet, Chairman;
John F. Nugent,
Charles W. Hunt, (Commissioners)
Huston Thompson,
William E. Humphrey,

In the Matter of	:	Docket 1233
PERMUTIT COMPANY	:	ORDER OF DISMISSAL

The above-entitled proceeding coming on for consideration upon the complaint of the Commission, answer of respondent, testimony and evidence, briefs and argument, and the Commission now being fully advised in the premises,

IT IS ORDERED that the complaint herein be and the same is hereby dismissed.

By the Commission:
Otis B. Johnson, Secretary.

NATIONAL FAST LIGHT BLUE

The National has just brought to the attention of dyers a new, bright blue for wool designated National Fast Light Blue BL. This new addition to the National line is said to be distinguished for its low cost and extreme fastness to light, qualities that recommend it for wide use on almost all classes of fabrics made of wool where economy and fastness to light are essential.

This new product, it is also claimed, possesses extreme solubility, dyes level, and embodies the further good qualities of fastness to water, stoving, hot pressing, decatizing and rubbing.

It is recommended as a self-color for bright shades of blue, or for use in combinations with other acid dyes of the fast to light group for the production of compound and mode shades on all classes of wool fabrics where fastness to light and economy are matters of first consideration, as in the coloring of women's wear fabrics, floor coverings, draperies, etc.

AMERICAN
DYESTUFF REPORTER

Devoted to the use and application of dyestuffs and the mechanical equipment incidental thereto.

LOUIS A. OLNEY
PROFESSOR OF CHEMISTRY AND DYEING, LOWELL TEXTILE SCHOOL
Directing Editor

CLAYTON HOAGLAND
Managing Editor

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THE DELUSION OF DYE ECONOMY

IN announcing its intention to start an inquiry to determine the average costs of dyestuffs entering into finished fabrics and garments, the Tariff Commission is stirring up interest in a topic that has never been brought with sufficient emphasis to the attention of those who should be most concerned with it. To textile manufacturers any reliable data bearing upon the cost of the dyes they use in their fabrics in relation to the costs of other items of mill production *ought* to be of particular interest.

But man's nature seems never to be complete without a few specious fallacies and delusions of one sort or another—particularly when such fallacies serve to excuse practices that might otherwise appear quite inexplicable. Textile men have frequently deluded themselves into believing that the cost of dyestuffs contributes to a considerable extent toward the total cost of producing the finished goods. The findings of the Tariff Commission's inquiry will prove beyond all question that the money spent for dyes is an insignificant item in mill production costs.

Whether or not the Commission's figures will puncture the delusion depends entirely upon the attitude in which those figures are accepted by the textile manufacturer. When it is shown in plain dollars and cents—most likely cents—that the cost of the dye, in its ratio to the total cost of producing a given piece of fabric, is about as appreciable an item as the cost of the alcohol in a glass of one-tenth of one per cent, the fabric makers *ought* to see dyestuff prices in a different light. If they are buying the highest types of fast colors it should convince them that the few cents extra they pay for them is really a sound investment in prestige. If they are buying cheap, second-rate dyes for first-rate fabrics it should convince them that they can easily afford to *double* their expenditures for this item, if for no other reason than for the prestige inherent in an honest, money-back, fast-to-light-and-washing guarantee.

In these times of consumer demand not only for fabric quality but for fastness qualities; in these times of low prices of American dyes, what excuse can any fabric manufacturer offer for using inferior dyes in fabrics for which light or washing fastness is an important requirement? Is it not an established axiom that the fastness or fugitiveness of a fabric will be remembered long after the price has been forgotten? Save money on overhead, cut down production costs, employ an efficiency expert—but why economize on the one item that *sells* the goods and strengthens reputation in the trade, when that item appears on the ledger in figures that make a dent in the column of dollars? Nothing can satisfactorily explain such a practice except the aforementioned fallacy that the cost of dyestuffs is responsible for a larger share of production outlay than it actually is; and this, too, in apparent disregard of Government figures which show dye prices slashed in half during the past four years—the weighted average slumping from $1.08 in 1920 to 0.54 in 1924 and Indigo and other bulk colors floating along the bottom of the market.

There is something very convincing about Government figures and we only hope that the findings of the Tariff Commission in its dye cost inquiry will convince some textile manufacturers that if they would enhance the prestige of their products in the trade it is high time to stop buying dyes with an eye to economy and stock up on the "real expensive" colors. Perhaps if dye costs were calculated on the basis of *cost per pair of hose or garment* instead of on cost per pound of dye these manufacturers would convince themselves before the Tariff Commission completed its data. In any case, the sooner the better.

AIR MAIL SPEEDS BUSINESS

MEANS of speedy communication are vital to the business world, and the recent installation of Air Mail Service is the most useful and important measure of postal improvement since the adoption of the railway postal car system in the sixties. The effect of the new Air Mail routes upon the postal service of the country as a whole should therefore be clearly understood by the business world.

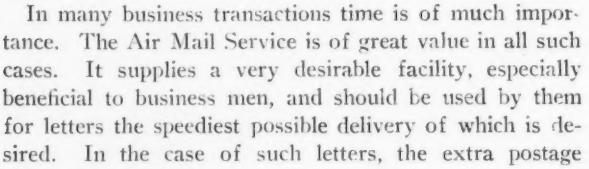
Transmission of mails by air is effected in less than one-half the time required by train. Air Mail letters dispatched from New York to-day about 9 p. m. are delivered at their address in Chicago by first carrier delivery to-morrow morning; or if forwarded from Chicago by train to other points, will move by the first morning train instead of by a late evening train. The delivery of such letters in postal territory served from Chicago will thus be expedited by at least twelve hours and often much more.

A similar gain in time is made with respect to other Air Mail stations where mails are transferred to railroads for distribution in adjacent territory.

Thus, a very wide zone on either side of the Transcontinental Air Mail route, letters destined to any point

within that zone may be expedited in delivery by several hours.

In many business transactions time is of much importance. The Air Mail Service is of great value in all such cases. It supplies a very desirable facility, especially beneficial to business men, and should be used by them for letters the speediest possible delivery of which is desired. In the case of such letters, the extra postage charge is negligible.



RECENT LITERATURE

Notes on the Practical Dyeing of Celanese. Notes on the Treatment and Dyeing of Celanese by the Garment Dyer. American Cellulose & Chemical Manufacturing Company, Ltd., New York.

Two instructive technical pamphlets on the use of SRA colors on Celanese. The first embraces information as to pretreatment of Celanese and mixed goods; dyeing materials; dyeing Celanese and mixed goods with SRA colors, and basic colors; and appends a number of dyeing recipes for Celanese alone in fabric or yarn; for Celanese and cotton, woven piece goods in solid shades and bicolor effects; and for hosiery; with a list of cotton colors for Celanese resist purposes and a brief word about Celascour, a new colloidal liquid preparation specially devised for use in scouring and dyeing goods containing Celanese yarns.

The pamphlet for the garment dyer is essentially similar but has in addition a section on dyeing Celanese and wool or real silk by the two, and one bath methods and on after-treatment, with a list of wool colors for Celanese resist purposes suitable for dyeing from acid and neutral baths, and a brief note on the selection of solvents for dry cleaning Celanese.

Textile Library—Vol. VII: Modern Dyeing Appliances; Part II: Mercerizing Cotton. Part II: Mercerizing Yarns. By Dr. John H. Haerry. The Textile Library Company. 279 pages. Illustrated.

Another volume in the Textile Library series of technical books on modern dyeing, bleaching, finishing, equipment and machinery. These books constitute a veritable encyclopedia of authoritative information about the construction and operation of the textile processing machinery of various American and foreign manufacturers. In this volume discussions of two distinct subjects are continued from previous issues.

The second installment of Modern Dyeing Appliances takes up jigs of various construction, padders, continuous dyeing machines, warp dyeing machines and other similar types, and is well illustrated with photographs and diagrams of this equipment. It includes an article on "The Relation of Monel Metal to Dyeing Machinery and to Textile Machinery in General" with sections on hosiery and knit goods machinery, yarn and skein dyeing machines, raw stock dyeing, bleaching equipment, etc.

The second installment of Mercerizing Cotton, first continuing the discussion of various methods of mercerizing piece goods, takes up in detail the rolling pressure method, modern tenter mercerizing ranges, chainless piece mercerizing machines and their operation, and other types of mercerizing machinery. Part 2 then follows, dealing with the mercerizing of yarns. Its chapters describe at length the construction and testing of yarn, skein mercerizing, preparation of yarn for mercerizing, and the actual mercerizing process, with a description of the machinery used; warp yarn mercerizing; yarn mercerizing without tension; caustic soda and its recovery; and tests for mercerized cotton. Copiously illustrated, the text is clear and highly instructive.

Organic Chemistry in the Hands of the Physician and the Lawyer. By Charles H. Herty, Ph.D. The Society of Medical Jurisprudence. 16 pages.

A paper presented before the Society of Medical Jurisprudence at one of its regular meetings at the New York Academy of Medicine, October, 1924, with the discussion that followed. Dr. Herty, with characteristic simplicity and animation, shows the close relation of industrial chemistry to the work of the man of medicine and the man of law, with a plea for the co-operation of chemistry, physics, pharmacology and biology. He briefly traces the rise of the American synthetic organic chemical industry and its place in our economic system.

The Future of the Synthetic Organic Chemical Industry in America. By Charles H. Herty, Ph.D. Synthetic Organic Chemical Manufacturers' Association. 14 pages.

A paper read before the Division of Industrial and Engineering Chemistry, American Chemical Society, April, 1925. In this comprehensive address Dr. Herty reviews progress in coal tar and non-coal-tar products, lead tetraethyl, pyrotechnic developments, new medicines and petroleum products; discusses research, problems in chemical education and competitive conditions; warns of the danger of foreign loans; and tells of the importance of chemical patents in their relation to a soundly constructed industry.

Both this booklet and the one reviewed above may be obtained from the Synthetic Organic Chemical Manufacturers Association, 1 Madison Avenue, New York City.

Dyeing Artificial Silk Fabrics. Rodney Hunt Machine Company, Orange, Mass. 40 pages.

To the average textile chemist the task of keeping well informed upon developments in artificial silk processing is assuming a most discouraging aspect. The literature on artificial silk is increasing in quantities far out of proportion to the interest in the fiber itself, no matter how great this latter may be. It is plainly impossible to read every word that has so far appeared on the subject since its comparatively recent rise to a place in the industrial limelight. And we shall not hear the last word for many years.

Yet there appears now and then concisely written treatises on some specific phase of artificial silk manufacture that cover more ground in a few pages than similar attempts cover in a volume. In this class is "Dyeing Artificial Silk Fabrics," published by the Rodney Hunt Machine Company. It is a pocket-size booklet of 40 pages bound in green artificial leather. In addition to much general information on the kinds of artificial silk, it treats instructively of dyeing the various types of this fiber, or methods of handling fabrics in each class during processing, of preparation for dyeing and of drying and finishing, with useful suggestions on the most practical procedures in the last two operations. The section dealing with important points to be considered in the selection of dyeing and finishing machinery for artificial silk supplies information that much of the literature on the subject has neglected.

This booklet is the first in a series which will be published under the general title of "Modern Practice in Textile Wet Finishing."

Third Progress Report of the Sub-Committee on Benzol. Chemical Section, National Safety Council. Published by National Bureau of Casualty and Surety Underwriters.

A fifteen-page report that was presented before the Cleveland Safety Congress, September 28 to October 2, 1925, containing the history of the investigation on benzol poisoning and the findings of the committee, with the results summarized in tabulated form.

1925 *Year Book of the National Association of Cotton Manufacturers*, combined with the *Cotton Manufacturers' Manual.*

The eighth edition of this year book, recently published, will, like its predecessors, be found of great value and interest not only to cotton manufacturers but to many others interested in the textile industry. The publication consists of three specific parts, the first dealing purely with the matters of the association, including lists of officers, members and by-laws of the association. The second portion is comprised of approximately 150 pages of statistical material which gives valuable information in regard to cotton production, consumption, exportation and cost, not only for the past year but over a period of years. The third section is of a technical character and contains many tables relating to weight equivalents, counts of yarn, humidity charts and various conversion tables.

The following new subjects have been added to the 1925 Year Book: Average Yarn Sizes for Knitting Machines; Allowance for Stops on Knitting Mill Machinery; Table Showing the Number of Slots in Cylinders; Latch Needle Gauge and Needles per Inch; Production of Cotton Knit Underwear; Average Underwear Production; Standard Textile Test Methods for Fabrics and Yarns; Correction Tables for Converting the Apparent Strength to a 6½ Per Cent Basis for Sheeting and Osnaburg; Standard Measurement of Hosiery Sizes; Standard Size of Bed Blankets; Standard List of Wide and Sail Duck.

CHEMICAL INDUSTRY IN RED CROSS DRIVE

The chemical and dyestuff trades have been mobilized for the Ninth Annual Roll Call of the American Red Cross. Harry Stebbins, general manager of Powers-Weightman Rosengarten Company, will head the manufacturing chemists; H. A. Metz, president, General Dyestuff Corporation, colors and dyestuffs; William Sanson, Knickerbocker Cleaning & Dyeing Company, cleaners and dyers, and Percy Magnus, of Magnus, Maybee & Reynard, essential oils.

The drive began on Armistice Day, November 11th; 5,000 volunteers will cover the industrial life of the city, asking people to volunteer their dollars so that the Red Cross may be kept on the line of duty ready for service day or night.

Each chairman will be assisted by a special committee of trade representatives and will co-operate with the Citizens Committee of 1,000 under the chairmanship of General James G., Harbord, president of the Radio Corporation of America.

The need for funds this year is more urgent than ever because of a 64.1 per cent increase in work for ex-service men, bearing out the prophecy of Surgeon-General Ireland who predicted five years ago that "the peak of the work for the sick and disabled ex-service men will not be reached until 1926."

Approximately 50 per cent of the annual expenditure of the New York County Chapter and the Bronx is devoted to post-war service.

CIBA TRANSFERS COLUMBUS OFFICE TO ATLANTA

The Columbus, Ga., office of Ciba Company was transferred on November 1st to Rhodes Building, 78 Marietta Street, Atlanta, Ga.

Under the management of George G. Wallace, formerly of Columbus, Ga., customers of Ciba will be offered a more complete service as a service laboratory and warehouse will be maintained at this point.

Dyestuff Tables

A List of the Principal American-Made Colors with Their Properties and Fastness Characteristics

[NOTE: The publishers of The REPORTER have secured more or less complete data in regard to many of the dyestuffs which are at the present time being manufactured in the United States. As the following material has been brought together from various sources, they realize that errors are quite likely to appear, and it is earnestly requested that any readers who find errors or who are able to give additional information will communicate with the publishers in order that this material may be made as correct, complete and up to date as possible. These tables will appear serially until all colors upon which we have data shall have been covered. The publication of this data was begun in the issue of March 10, 1924.]

CORRECTION

In the Dyestuff Tables appearing on page 707 of the October 19 issue the name of the Newport Chemical Works, Inc., was, through a regrettable oversight, omitted as an American manufacturer of Indanthrene Golden Orange G and Indanthrene Golden Orange RRT. The Newport Chemical Works produces both of these vat colors, the first under the name of Anthrene Golden Orange G and the second under the name of Anthrene Golden Orange RRT. Readers who are preserving these tables should make note of this correction under these two products.

ANTHRENE RED BN

(Schultz No. 831)

SPECIALLY SUITABLE FOR: Cotton.

USUAL METHOD OF DYEING: Must be dyed cold with large amount of Glauber salt. Use 2½ to 30 per cent of dye, 2½ to 8 per cent of hydro, 2 per cent dry caustic and 10-80 per cent Glauber salt. If bath is cloudy add more caustic.

SHADE: Brighter than Indanthrene Red R (S-830).

FASTNESS TO:

Acid: Fast.
Alkali: Fast.
Chlorine: Fast.
Cross-Dyeing: Fast.
Ironing: Fast.
Light: Excellent; best of the vat reds.
Potting: Fast.
Rubbing: Fast.
Steaming: Fast.
Sulphur: Fast.
Washing: Fast.
Water: Fast.
Bowking: Slight.

MACHINE DYEING: Suitable.

DYED BY OTHER METHODS: Can be pad-dyed.

ON OTHER MATERIALS: Wool: Bluer and duller than Alizarine Red (S-780) on an alumina mordant; fast to washing, fulling, dry steaming, stoving, alkalies and carbonizing; very fast to potting, cross-dyeing and light. Silk: Unsuitable; dyes a pale pink.

PRINTING: Steaming process without soda fixation is best. Printed shade is faster to light than S-826 or S-830.

DISCHARGING: Rongalite CL a white.

COMPETING PRODUCTS: Made in the United States by Newport Chemical Works, under name of Anthrene Red BN.

SOLAMINE BLUE FF

(No Schultz Number)

SPECIALLY SUITABLE FOR: Cotton.

USUAL METHOD OF DYEING: Glauber salt and soda ash.

SHADE: Much brighter than S-456.

SOLUBILITY: Good.

LEVEL: Dyes level.

EXHAUSTS: Well.

FASTNESS TO:

Acid: Turns redder with dilute sulphuric.
Alkali: Fast.
Chlorine: Destroyed.
Ironing: Turns redder.
Perspiration: Fast.
Washing: Moderate.

ON UNIONS: Cotton-silk unions: Dyed with Glauber salt and soda ash, the cotton is darker than the silk. Cotton-wool unions: The wool is stained slightly reddish.

COMPETING PRODUCTS: Made in the United States by Newport Chemical Works, under name of Direct Fast Blue SFF; Cincinnati Chemical Works, under name of Direct Fast Light Blue FF; E. I. du Pont de Nemours & Co., under name of Pontamine Fast Blue SLN; National Aniline & Chemical Company, under name of Niagara Fast Blue RL.

KROMEKO GREEN B

(No Schultz Number)

COMPOSITION: Identical with Anthracene Chromate Green B.

SPECIALLY SUITABLE FOR: Wool.

USUAL METHOD OF DYEING: Chromate.

SHADE: Yellower and duller than Diamond Green SS.

LEVEL: Moderate.

EXHAUSTS: Moderate.

FASTNESS TO:

Acid: Fast.
Alkali: Fast.
Carbonizing: Fairly good.
Fulling: Fast.
Light: Better than the Diamond Greens.
Potting: Fast.

Rubbing: Fast.
Steaming: Fast.
Sulphur: Fast.

SENSITIVE TO METALS, LIME: Sensitive to metals. By dyeing chromate this sensitiveness is reduced.

DYED BY OTHER METHODS: Can be dyed top or bottom chrome. Top chrome is faster to potting.

ON UNIONS: Cotton-effect threads are tinted reddish.

PRINTING: Suited only for vigoureux. Bleeds greenish into white. The prints with fluoride are faster to hot water than those with acetate.

COMPETING PRODUCTS: Made in the United States by Amalgamated Dyestuff & Chemical Works (Selling Agents, John Campbell & Co.).

NEROL 2B

(Colour Index No. 304)

SPECIALLY SUITABLE FOR: Wool; especially knitting yarns.

USUAL METHOD OF DYEING: Glauber salt and a very little acetic acid.

SHADE: Blue black.

SHADE BY GASLIGHT: Grayish.

SOLUBILITY: Good.

LEVEL: Dyes level with care; sensitive to acid.

EXHAUSTS: Well.

FASTNESS TO:
Acid: Fast.
Alkali: Fast.
Carbonizing: Fast.
Fulling: Fast.
Ironing: Fast.
Light: Moderate.
Perspiration: Fast.
Sulphur: Rather moderate.
Washing: Very good.
Finishing: Excels Sulphoncyanine Black.

SENSITIVE TO METALS, LIME: Sensitive to lime, copper tin or iron. Not sensitive to chrome.

ON UNIONS: Dyes wool neutral better than S-266. Cotton effect threads stained.

ON OTHER MATERIALS: Silk: Suitable. Leather: Suitable on vegetable and chrome-tanned.

PRINTING: Fast to water and soap.

COMPETING PRODUCTS: Made in the United States by Newport Chemical Works, Inc., under name of Fast Acid Black N2B; Garfield Aniline Works, under name of Sulphon Acid Black N2BM.

ALIZARINE EMARADOLE G

(Colour Index No. 1056)

SPECIALLY SUITABLE FOR: Wool.

USUAL METHOD OF DYEING: Acid.

SHADE: Duller than Acid Green 6B.

SHADE BY GASLIGHT: Brighter and clearer.

SOLUBILITY: Good.

LEVEL: Dyes level, but inferior to SAP.

EXHAUSTS: Well.

FASTNESS TO:
Acid: Fast.
Alkali: Fast; somewhat bluer with soda.
Carbonizing: Fast.
Fulling: Fair in a light milling.
Light: As a self-shade, moderate. In mixtures, considerably better than Acid Green 6B, Patent Blue or Brilliant Acid Green 6B.
Perspiration: Fast.
Rubbing: Fast.
Sulphur: Moderate; becomes considerably bluer. The shade returns when acidulated.
Salt Water Spotting: Equals Saphirol SE.

SENSITIVE TO METALS, LIME: This color is of the same type as Alizarine Saphirol (S-858) and Alizarine Astrol. It is not as level dyeing nor as fast to light as Saphirol.

OTHER PROPERTIES: Principally for compound shades on ladies' dress goods.

DYED BY OTHER METHODS: It is much faster to light in compound shades than as a self-shade.

ON UNIONS: Cotton-effect threads remain white.

ON OTHER MATERIALS: Gloria: The silk is lighter than the wool; with acetic acid the silk is left white. Silk: The fastness to water on this fiber is moderate.

PRINTING: May be used for printing woolen pieces and for vigoureux. When printing sour a bluish green is obtained With fluoride and acetate of chrome a yellowish green. Prints with acetate of chrome turn out considerably weaker. The fastness of the acid prints on slubbing is only fair. The fastness to fulling, however, is improved by chrome.

COMPETING PRODUCTS: Made in the United States by E. I. du Pont de Nemours & Co., under name of Alizarine Emarald; Grasselli Dyestuff Corporation, under name of Alizarine Emaradole G.

ALIZARINE RUBINOLE R

(Colour Index No. 1091)

SPECIALLY SUITABLE FOR: Wool.

USUAL METHOD OF DYEING: Sulphuric and Glauber salt.

SHADE: A trifle yellower than Azo Fuchsine 6B.

SHADE BY GASLIGHT: Turns yellowish.

(Continued on page 779)

RECENT PATENTS

Bleaching Process

(1,547,138; July 21, 1925)

John R. MacMillan, of La Salle, New York, assignor to Niagara Alkali Company, of Niagara Falls, New York, a Corporation of New York.

This invention relates to bleaching processes; and it relates to a method of bleaching fiber wherein such fiber is treated with a modicum of bleaching solution (hypochlorite) and with chlorine and finally with enough more bleaching solution to accomplish the degree of bleaching desired; such process being particularly applicable to ligniferous fibers rich in lignin, such as jute, but also applicable to other fibers; all as more fully hereinafter set forth and as claimed.

In bleaching all sorts of fibers and fabrics it has of course long been the practice to treat the fiber with a bleaching liquor containing a hypochlorite; this bleaching liquor containing hypochlorite of calcium, of sodium, or of potassium as the case may be. A solution of bleaching powder (which contains calcium hypochlorite) is often used. Sometimes bleaching liquor is made by chlorinating a caustic soda solution. Afterwards the fabric is washed, etc., and is, in some cases, treated with an antichlor. This method of bleaching has been known and used for many years. For some purposes, as in bleaching paper pulp, it has been found more convenient to use a preliminary treatment with chlorine before treating with the regular bleaching liquor. Part of the bleaching is done by the chlorine and the rest by the bleaching liquor.

With many fibers there is a practical limit to the extent to which bleaching can be carried in these ways, this being particularly true with fibers, like jute, rich in lignin. With such fibers production of a full white or complete bleaching is attended with too much loss in weight or too much "tendering"; that is weakening of the fiber.

Much better results can be obtained by a special method of bleaching whereby it is possible to obtain an equal degree of bleaching with considerably less bleaching agent or, in using the same amount of bleaching agent a much greater bleaching result can be attained, and with the further unexpected result of a lessening in the usual loss of weight and tendering, thereby making it possible to secure a full bleach in some fibers, like jute, usually regarded as too refractory for a white bleach. Furthermore in this special method several other results are accomplished which do not follow from the use of the ordinary methods; these results being a diminution in the total ash of the fiber and a diminution in the amount of ether extract—the latter representing fats, resins, etc. Sometimes the reduction in ash and ether content by the new method is astonishingly great. In one particular case a jute with about 14 per cent total ash, bleached by the ordinary methods still carried 10 per cent ash while when treated by the present method and completely bleached it carried but 2 per cent.

From the facts, it appears that, although ordinary bleaching agents (chlorine and bleaching liquor) are used in this method and an unusually effective bleach is secured, the agents are doing something more than bleaching and, since the extra effects are particularly noteworthy with fibers rich in lignin, that the extra effects are due to reaction with non-cellulose components of the fiber other than lignin. Whatever the reason, the facts are as stated.

In the present invention the fiber is first treated with a small amount of bleaching solution, this bleaching solution being made from bleaching powder in the ordinary way or in any other way, as by leading chlorine into a solution of alkali. The liquor is, as is usual with bleaching liquors, slightly alkaline. The amount used on the fiber is usually around 10 per cent of the total amount of bleaching liquor which would be required to effect a substantial commercial bleaching action; in other words, the amount here used is, from the bleaching standpoint, extremely small. As soon as the bleaching liquor is incorporated into the pulp it is treated at once with gaseous chlorine, blowing it into the pulp or otherwise contacting it with the fiber to be treated. In working with pulped fiber take a 3 to 5 per cent pulp, stir the required amount of bleaching liquor into this, and then blow chlorine into the pulp for the time necessary to effect the action desired—say, about ten to fifteen minutes—with a slow introduction of chlorine. In operating in this way, the material is attacked practically simultaneously by an alkaline bleaching liquor and by a solution of chlorine; this solution being, in large part, of chlorine as such. Chlorine on contact with water does not hydrolyze into hypochlorous and hydrochloric acids at once, but remains for a time as a solution of chlorine itself. And when hydrolysis occurs, it goes to a balance or a dynamic equilibrium between unhydrolyzed chlorine and its hydrolyzed reaction products, hypochlorous and hydrochloric acids. In the presence of hypochlorite (added as bleaching liquor) this hydrolysis is correspondingly retarded. The net result in the present process is that for a time the fiber is exposed both to the action of a hypochlorite, hypochlorous acid, and to that of chlorine itself; something which seems to be necessary in accomplishing the present results. In these actions the non-fibrous constituents, etc., which encrust the fibers are attacked and either oxidized or rendered water-soluble or alkali-soluble. After the requisite amount of chlorine has been introduced, this amount being usually equal to about 50 per cent of the total bleaching agent required, the fiber is washed, in certain cases the slight amount of hydrochloric acid present is neutralized with caustic soda or lime, and the fiber is finally put through the usual bleach with bleaching liquor; the amount of bleach used being diminished, however, to allow for the bleaching agent used in the two prior stages. For an equal degree of bleaching, the total amount of bleaching agent used in

the three bleaching steps of the present process is considerably less than by either of the older methods hereinbefore described; or, for an equal amount of bleaching agent considerably more bleaching is effected. And in some cases, and particularly with ligniferous fibers, as with the jute mentioned, with kraft pulp, etc., bleaching results can be accomplished which are impossible by any of the known methods; that is, without undue loss of weight and tendering.

Most vegetable fibers contain cellulose and, in addition, the bodies which collectively are known as lignin, the proportion of lignin and cellulose varying from fiber to fiber. Some fibers, like cotton, contain very little lignin, while others, like jute, are rich in lignin and relatively poor in ecllulose. Lignin is considerably more reactive chemically than is cellulose; a fact which is responsible both for the loss of weight and the tendering occurring in any drastic bleaching of fibers rich in lignin.. Chlorine attacks lignin-forming products soluble in alkaline solution, and the lignin is, moreover, susceptible to oxidation. In the usual methods using chlorine, any attempt to secure far-going bleach of jute results in a great loss of fiber, in tendering, etc. But, on the other hand, although chlorine is used in the present method, the loss of fiber with fibers rich in lignin is not greater than in usual methods of treating fibers poor in lignin, such as cotton, sulphite pulp and the like, and the bleaching action is sufficient, while there is no tendering or weakening of the fiber. Bleaching liquor and chlorine acting together in very dilute solution, as is the case in this method of bleaching, for some reason do not exercise the deleterious effects of either, as is the case at the higher bleaching concentrations usually employed. The activity of this combination of reagents is great, but it specifically attacks the coloring matter and encrustants which are relatively more susceptible to their action rather than the cellulose or the fiber structure. For this reason the bleaching action is more effective than when either is used separately at the higher concentrations usually employed.

TWO NEW RHODAMINES

Two new Rhodamines have just been announced by E. I. du Pont de Nemours & Co., under the names Du Pont Rhodamine 5GDN Extra and Du Pont Rhodamine 6GDN Extra.

These two are similar types of Rhodamines which differ only to a small degree in yellowness of shade. They are said to be especially useful for color discharge in textile printing as, it is claimed, they are only slightly affected during the steaming operation by the reducing agent employed for the discharge.

These colors are also used for printing cotton, silk, wool, etc., and for dyeing whenever a bright yellowish pink shade is desired. They are described as fast to stoving and can be used for obtaining bright shades on wool which is to be bleached after dyeing.

NEW CHROMOTOL COLOR CARD

Dunker & Perkins Company has just brought out a new color card showing the complete line of Chromotol colors.

All these colors are dyed with the addition of acetate of ammonia and chrome to the dye bath. They are said to exhaust well but for the heavier shades it is recommended that the dye bath be cleared with an addition of acetic acid.

All the colors, it is claimed, possess good fastness to light and fulling, and are especially recommended for the colorings of woolens and worsteds for ladies' and men's wear.

It is said furthermore that piece goods or yarn dyed by this method are left in much better condition than when dyed by the top or bottom chrome method.

The new color card may be obtained by request to Dunker & Perkins Company, 263 Summer Street, Boston.

TWO NEW ANTHRENE COLORS

The Newport Chemical Works, Inc., has issued leaflets describing the dyeing properties of two new additions to its line of Anthrene colors, Newport Anthrene Black DS and Newport Anthrene Scarlet 2G paste. The leaflets also contain swatch bows exhibiting these new colors, the black on pure silk and the scarlet on cotton and rayon.

Newport Anthrene Black DS is a direct dyeing vat black primarily intended for dyeing silk. It is also of value for dyeing rayon, on which fiber it produces a bright bluish shade of black. No special after-treatment is required to develop the shade. It is said to dye very level, penetrates well and possesses excellent fastness to the usual color-destroying influences. It is not suitable for either Lustron or Celanese.

Newport Anthrene Scarlet 2G Paste is described as of great interest for dyeing cotton, rayon and pure silk fabrics. In addition to its value for dyeing purposes is also of particular interest for printing cotton material. Bright shades of scarlet are obtained which are said to be distinguished by excellent fastness to washing, chlorine and very good fastness to light. It is, according to the leaflet, easily reduced and possesses good level dyeing and penetrating qualities.

CIBA ANNOUNCES NEW NEOLAN COLOR

An important addition to the group of Neolans is announced by Ciba Company, Inc. In Neolan Yellow GR they introduce a color which is said to produce exceptionally fast to light and milling shades from an acid bath. All other properties which come into question in dyeing wool fast are also very good.

Neolan Yellow GR, it is claimed, levels very well, and is therefore suitable for combination with other Neolans for the production of the fastest piece goods. Neolan Yellow GR is especially suitable for slubbing and loose wool. This new color possesses better tinc-

torial strength and leveling properties than the old marks, Neolan R and B.

This new Neolan color is also suited for ordinary and weighted silk, such dyeings, according to the announcement, being exceptionally fast to light, water, washing and perspiration. Neolan Yellow GR is also recommended for wool and silk printing, with or without the addition of chrome acetate.

FOUR NEW DU PONT PRODUCTS

The dyestuffs department of E. I. du Pont de Nemours & Co. announce the addition to their line of chrome blacks, of Du Pont Alizarine Blue Black B, the most characteristic property of which is said to be excellent fastness to light, even in very delicate shades.

In addition, it is said to be very soluble and to dye very evenly and penetrate well; consequently its chief use is for grays, light blues, etc., on men's wear materials of various types.

It has the usual very good fastness qualities of the chrome colors, according to the announcement, and can be applied by either the top chrome, bottom chrome, or chromate process with good results, although for most purposes it gives the best results by either bottom chrome or chromate method.

A new vat green also announced by this concern is Ponsol Brilliant Green G Paste, which, it is claimed, produces on cotton shades fast to chlorine, washing and light.

By combination with other colors of the vat series possessing similar exceptionally good qualities, it is possible to produce a series of bright greens from the bluest to the yellowest tones.

This product is suitable for cotton goods in any stage of manufacture and is especially desirable for such materials as shirtings, towelings and dress goods which require good fastness to chlorine, washing and light. It can be applied to piece goods either on the padder or the jig, and when properly applied is useful for silk and rayon.

Two new basic colors that have just been added to the Du Pont line, Du Pont Rhodamine 6GDN Extra and Du Pont Rhodamine 5GDN Extra, are offered especially for the printing trade for bright yellowish pinks on wool, silk and cotton.

They are particularly recommended for color discharge work on all fibers, as they are said to be unaffected by this discharge process, and to retain their brilliancy.

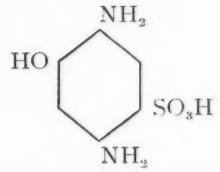
They are also offered for dyeing bright yellowish pinks on silk, rayon, cotton and wool. On silk they display a pleasing fluorescence. They are especially suitable for stripes in woolen blankets and other materials which are to be subsequently bleached, as they are not affected by this process. The fastness to washing is also sufficiently good for such use.

The two products are identical in properties, but Du Pont Rhodamine 5GDN Extra is bluer in shade.

THE REDUCTION PRODUCTS OF AZO DYES

(*Continued from page 754*)

No. 39.—2, 5-DIAMINOPHENOL-4-SULFONIC ACID

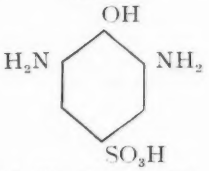


$C_6H_8N_2O_4S$; Mol. Wt. 204.17; (C, 35.3%; H, 4.0%; N, 13.7%; O, 31.3%; S, 15.7%).

Derived from: C. I. 409.

Apparently no data is available on this compound.

No. 40.—2, 6-DIAMINOPHENOL-4-SULFONIC ACID



$C_6H_8N_2O_4S$; Mol. Wt. 204.17; (C, 35.3%; H, 4.0%; N, 13.7%; O, 31.3%; S, 15.7%).

Derived from: C. I. Nos. 336 and 337 and by the complete reduction of 172.

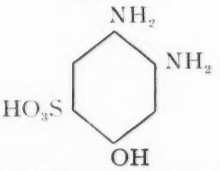
White cryst. which gradually darken in air.

Its sols. are colored deep brown by oxidizing agents.

Readily diazotized giving a very stable, readily sol., deep yellow tetrazo deriv., which forms a blue-black dye with β-naphthol, violet in aqueous sol., turned red by mineral acids.

The characteristics of a considerable no. of disazo dyes, obtained by coupling with two different components, are given in D. R. P. 148212 (Friedl. VII, 393).

No. 41.—3, 4-DIAMINOPHENOL-6-SULFONIC ACID

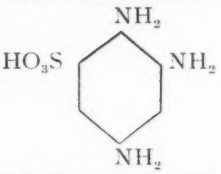


$C_6H_8N_2O_4S$; Mol. Wt. 204.17; (C, 35.3%; H, 4.0%; N, 13.7%; O, 31.3%; S, 15.7%).

Derived from: C. I. No. 602.

Apparently no data is available on this product.

No. 42.—1, 2, 4-TRIAMINOBENZENE-5-SULFONIC ACID



$C_6H_9N_3O_3S$; Mol. Wt. 203.19; (C, 35.4%; H, 4.5%; N, 20.7%; O, 23.6%; S, 15.8%).

Derived from: C. I. Nos. 98 and 352A.

Apparently no data is available on this product.

Technical Notes from Foreign Sources

Minute Structure of Fibers

Studies of the X-ray diagrams of cellulose in monochromatic light shows certain deviations and divisions of the points which are only explained by the existence of more than one crystalline substance. The concentric layers of bast fibers show under the microscope parallel fibrils. Naegeli's miscellae, which possess an inclination to the axis which is characteristic for each layer. The bast fibers of hemp, mulberry, and broom give distinct fiber diagrams which comprise a complex of layers.

The ramie fibers show two complexes of layers; flax is indefinite and cotton has a diagram which is characteristic of the spiral arrangement. Mercerization produces changes in the molecule of the constituent of cellulose which shows the maximum interferences, without sensibly affecting the size of the molecule. Artificial silk shows the same diagram as mercerized cellulose. Silk fibroin and chitin show a certain similarity to cellulose; the main body is either a single crystalline substance or a mixture of such.

The crystals lie with an axis in the direction of the fiber and are imbedded in a cementing substance. The elementary crystal particles are of similar dimensions to those of other organic compounds. The formation of fiber structure in the organism takes place through the crystallization of the amorphous substances in consequence of tension in the fiber axis, whereby the crystallographic direction of the greatest rapidity of growth lies in the fiber axis. The dementing substances are more readily attacked than the crystallites, since hydrocellulose, in spite of profound mechanical breakdown, shows the same diagram as normal cellulose. (Chem. Zent., 1925, volume 96, II, pages 132-3.)

Dyeing and Printing Textiles

The water soluble esters of the indigoid coloring matters which are described in British Patent No. 186,057, and which are stated in British Patent No. 203,681 to have no affinity for textile fibers are found to have a good affinity for animal fibers at high temperatures and are particularly useful for application to wool by the method which is generally used in the application of acid coloring matters, the true color of the indigoid coloring matter being developed afterward by means of suitable oxidizing agents. The application of these coloring matters to wool can thus be carried out entirely in acid liquors.

For example, wool is dyed an indigo shade by immersion for a period of thirty minutes at a temperature varying between 60 and 65 deg. Cent. in a dye liquor which contains a water soluble ester of indigo to the proportion of 1 per cent. One per cent and 2 per cent portions of a 96 per cent sulphuric acid are added at the end of successive periods of thirty minutes, and the temperature is maintained at a point varying between 80 and 85 deg. Cent. The dyed wool is afterward washed with water, then impregnated with a solution containing 20 to 30 grams of sodium nitrite per liter and developed in a solution containing 20 grams of 96 per cent sulphuric acid per liter at a temperature of 60 deg. Fahr., and finally the wool is washed and dried.

For further details see British Patent No. 218,649.

Usefulness of Detergents Containing Peroxides

As results that were obtained by comparison of the detergent values of washing powder containing sodium perborate, soap powder and soap in the removal of coffee, milk, wine, oil, blood, rust, ink (iron free) and particularly cocoa stains from cotton fabric confirm the author's previous conclusions that washing powders containing peroxides are more efficient than ordinary soap detergents and that such oxidizing preparations have no marked deleterious action on the strength of the fabric is being treated. Experiments in which the stained fabrics were rubbed with metalic copper and afterward cleansed in copper vessels indicates that there is little probability of the catalytic activity of copper causing damage to the fabrics during the ordinary processes of washing by means of detergents containing peroxides. (Chemiker Zeitung, 1925, pages 505-6.)

Printing Naphthol Black on Wool and Silk

Black shades which are obtained by the oxidation of Naphthol Black (Cassella) with a chlorate in the presence of vanadium are made faster to soaping and hot water by the addition of small amounts of Nitrophenamine G (Badische), and Paramine Extra to the oxidation mixture.

For example, a printing paste which is capable of yielding fast black shades is prepared by dissolving 200 grams of ammonium chloride in a mixture which is prepared by the addition of 700 grams of Naphthol Black BG which are dissolved in 3,000 c.c. of warm water to 50 grams of Nitrophenamine B, 50 grams of Paramine Extra, 1 liter of warm water and 4,200 grams of a 50 per cent aqueous solution of gum. Thereafter there is added 200 grams of sodium chlorate which are dissolved in 200 grams of water and then a mixture of 200 grams of a 0.1 per cent solution of lead nitrate and 200 grams of a 0.1 per cent solution of ammonium vanadate. The paste that is made in this manner is stable at ordinary temperatures. It is reported that the increased fastness is probably due to

the formation of fast brown pigments by oxidation of the Nitrophenamine G and Paramine Extra, and it is pointed out as well that the former substance is not being manufactured. (Bulletin Societe Ind. Muhlhouse, 1925, pages 322-3.)

Wool Effect on Cotton Fabrics

Within recent times the attention of the textile industry has been directed to a process which had for its purpose the obtaining of a wool effect on cotton fabrics which consisted in treating the fabric with nitric acid. According to the paper which is published in Melliand's Textilberichte, 1925, page 661, the same expectations are not to be assigned to this process as are obtained in the mercerization of cotton. For it remains that during the mercerization of the cotton fiber not only an improvement in the appearance of the textile fiber obtained but the strength of the fiber is increased as well. But in the case of the nitric acid process only an apparent increase in the strength of the cotton fibers is obtained. The shrinkage of the fabric during the treatment results, to be sure, is an increase in the absolute strength of the fiber, but after the nitric acid treatment the fibers remain colored a strong brownish yellow and it is necessary to remove this color by bleaching. But as may be gained from the observation that the nitric acid treated fibers possess strong reducing powers, it follows that the fibers are no longer as resistant to chemical influences. It therefore may be concluded, as is corroborated by actual tests, that a considerable reduction is effected in the strength of the cotton fibers by such after-treatments, as bleaching, dyeing, and printing.

It follows that the inherent properties of the fibers are therefore of importance as well as the manner in which they have been woven together to form a fabric, for in testing fabrics of different weaves very different results have been obtained. In order to have a correct conception of the change in the mechanical strength of the fibers it is necessary to take into consideration the shrinkage of the fibers, and it is best to refer the testing results to the tearing length at the time of testing, which means on the product of the strength of the fabric as determined by testing and the yarn number which has to be established. Comparison of the tearing length is not of importance to most practical men, for they are accustomed only to determine the direct strength factors without change in the number of threads per centimeter or contraction of the individual fibers being taken into consideration.

One conclusion that was derived from these tests was that the strength of the fibers varied very considerable according to the nature of the weave and other properties of the cotton fiber.

Preparation for Coloring Human and Animal Hair

Finely pulverized paraphenylenediamine is mixed with a concentrated solution of alkali polysulphites while the mass is constantly and very vigorously stirred. The operation is continued until the mass attain the properties of a salve. Thereafter the mass become solid after a short time. The preparation that is made in this manner according to the process which is patented in Swiss Patent No. 102,864, is said not to have the harmful action of paraphenylenediamine on human hair.

Use of Common Salt in Dyeing

Common salt is used in very large amounts and for a variety of purposes in the dyeing of textile fabrics. But it is not at all clear what the action of this chemical is in the dyeing process. For example, it is used in dyeing with the substantive coloring matters, and when it is employed for this purpose it is seen at once that the color bath is much better exhausted than when it is absent. This action of common salt depends on the simple fact that the coloring matter is much less soluble in a solution of common salt than it is in ordinary warm water, and hence it is more readily and more completely absorbed by the textile fibers which are dyed in this color bath.

In other cases, however, the action of common salt is of an entirely different character and, in fact, its action seems to be directly opposite to the above. For example, it often happens that a coloring matter is absorbed too rapidly by the textile fiber with the result that irregular dyeing results are obtained, in that parts of the fabric will be dyed before other have absorbed the color. The explanation of the action of common salt in this case is by no means as easy a matter. It appears that the action of the salt is to interpose itself between the molecule of the coloring matter and the fiber in such a manner that it slows up the combination between the two. It is just as though two persons wish to meet and are prevented doing so by a crowd of persons who come between them. If the salt really acts in this fashion, then large quantities of it must be present in the color bath, and at any rate the proportion of the salt in the color liquor must be greater than that of the colloring matter itself.

There is finally another role that is played by common salt in the dyeing process. It renders softer the various sizing preparations that are used in sizing textile fibers and that contain, as a general rule, both starches and gums. It is a fact that when these sizing compositions are employed without the addition of sodium chloride they lend the fabrics thus treated a rather harsh handle. The action of common salt in this case is to be assigned to the fact that there is always present a certain proportion of magnesium chloride in common salt and this ingredient makes the salt hygroscopic. The absorption of water by the sizing preparation that contains common salts makes it always a little soft so that the handle of the materials that are treated with it is never harsh and rough

The salt that is employed in the textile industries for these and other purposes must be free from metallic impurities as otherwise the fabrics are improperly dyed. (Deutsche Faerber Zeitung, 1925, page 1087.)

Washing Fast Printed Colors on Leather

In German Patent No. 411,365 there is described a process for the production of printed designs in color on leather goods which are fast to washing. The leather is first treated with formaldehyde, alum and the like, after-treated with vegetable mordants, such as sumac and then printed. After printing the leather is steamed in closed vessels and then worked up into finished form as Swedish leather and the like. The steaming process lasts one hour at a temperature of 75 deg. Cent.

DYESTUFF TABLES

(*Continued from page 773*)

SOLUBILITY: Very good.

LEVEL: Excellent; can be fed on at the boil.

EXHAUSTS: Clear.

PENETRATION: Excellent.

FASTNESS TO:

Alkali: Shade goes trifle bluer.
Carbonizing: Turns somewhat bluer; shade returns on neutralizing.
Fulling: Adapted for ladies' dress goods which are to be lightly fulled.
Light: Excellent; better than everything else in acid colors in this shade.
Rubbing: Excellent.
Steaming: Good.
Sulphur: Excellent.

SENSITIVE TO METALS, LIME: The R brand is not as well adapted for use with acetic acid as the G and 3G brands.

OTHER PROPERTIES: Largely used, as in borders, for white wool blankets.

DYED BY OTHER METHODS: On a chrome mordant or top-chromed, this color gives full shades which, however, are no faster than the acid dyeing. As this product is not affected by chrome, it is well adapted as a shading color for top and bottom chrome work.

ON OTHER MATERIALS: The dyestuff is adapted for dyeing silk, but it is not fast to water. On gloria, dyeings of uniform shade on both fibers may be produced. In a neutral Glauber salt bath wool is dyed well, while cotton threads remain white, or nearly so. In an acid bath cotton-effect threads are left pure white.

PRINTING: Well adapted for wool printing with acetic acid or oxalate of ammonia as fixing agents. Printed on silk, the color gives fine pink shades of good fastness to water. Not suitable for discharge printing.

COMPETING PRODUCTS: Made in the United States by Grasselli Dyestuff Corporation, under name of Alizarine Rubinole R.

ETHONIC BRILLIANT BLUE 2G

(No Schultz Number)

COMPOSITION: Identical with Azo Acid Blue 2G (Bayer).

SPECIALLY SUITABLE FOR: Wool.

USUAL METHOD OF DYEING: Glauber salt and sulphuric acid.

SHADE: Greenish blue.

SHADE BY GASLIGHT: Somewhat greener.

LEVEL: Inferior to S-63.

EXHAUSTS: Well.

FASTNESS TO:

Acid: Moderate dilute sulphuric and hydrochloric affect the shade.
Alkali: Fairly good; better than S-63.
Carbonizing: Shade returns on neutralizing.
Fulling: Moderate.
Ironing: Fast.
Light: Better than S-63.
Potting: Moderate.
Steaming: Turns somewhat greener.
Sulphur: Fast.
Washing: Fairly good.

SENSITIVE TO METALS, LIME: Very sensitive to acid; the shade turns a scarlet in contact with sulphuric acid or hydrochloric. After a good rinsing, however, the original shade returns. The color is very sensitive to chrome and a small amount will destroy it.

DYED BY OTHER METHODS: Goes well on wool in a neutral bath.

ON UNIONS: When dyed acid, cotton-effect threads are left white.

ON OTHER MATERIALS: Silk. The color gives a bright reddish-blue shade which is fast to water.

PRINTING: Well adapted for wool and carpet yarn printing with acetic. The fastness to water is fairly good.

DISCHARGING: Dischargeable with Rongalite C and tin salt, but the discharge is not a pure white.

COMPETING PRODUCTS: Made in the United States by Amalgamated Dyestuff & Chemical Works (Selling Agents, John Campbell & Co.).

ON THE TRANSPORTATION OF EXPLOSIVE AND POISONOUS ARTICLES

The Bureau of Explosives has recently issued a small pamphlet giving Interstate Commerce Commission regulations for the transportation of explosives and other dangerous articles by freight and express and as baggage.

including specifications for shipping containers. This booklet is actually only a supplement to the Bureau's "Pamphlet No. 9" and as such gives the *amendments* to the regulations contained in that pamphlet. The amended paragraphs are, in the supplement, printed in full, with the new matter heavily underscored.

GOVERNMENT TO STUDY DYE COSTS IN TEXTILE MANUFACTURE

An investigation to ascertain the average costs of dyestuffs used on fabrics and garments is to be undertaken by the United States Tariff Commission. A color specialist of the Chemical Division, Dr. W. N. Watson, will direct the inquiry and personally visit twenty-five textile mills in New York State and New England, as well as a number of dye manufacturing plants, in compiling the data upon which the average figures will be based.

The present inquiry, it is understood, will be limited to finding an average dyestuff cost for a few representative piece fabrics and garments. No attempt will be made to cover all types of apparel nor all types of dyestuffs used, although the work will include the commonly used fabrics, cotton, wool, worsteds, rayon and silk. A selected list of the bulk colors most widely used in piece goods and garment dyeing will comprise the groundwork for the cost data in checking the dyestuff sales prices with mill purchasing prices.

The calculations will cover the cost of dyeing certain classes of representative fabrics in the piece in connection with data compiled from the cutting-up trades as to the various qualities of such fabrics entering into a man's suit or overcoat. The average cost of dyestuffs used in hosiery will be included.

SCOURED WOOL MOISTURE TO BE CHARTED

A series of experiments have been started by the United States Departments of Agriculture and Commerce for the purpose of compiling standard figures on the moisture content of grease wool. These tests are part of the experiments being made by the Department of Agriculture in an endeavor to work out tables and charts indicating weight losses in scouring wool from various sections of the country. A wool-scouring laboratory has been established in Washington by the Department for this purpose.

In making moisture tests, samples of wool gathered from all parts of the country are placed in a specially designed drying oven, the samples being weighted both before and after drying. The oven, which is at the Bureau of Standards, is in an insulated chamber in which both atmosphere and humidity are artificially regulated.

Moisture in grease wool frequently runs up to 10 per cent by volume, declares D. W. Willingmyre, of the Department of Agriculture, and is an important factor in determining the price to be paid growers for their product. It is planned to work out a system as a result of these experiments that will enable wool buyers in the field to determine quickly the quantity of moisture in all given lots of wool.

DYE IMPORTS IN OCTOBER

Foreign dyes imported through New York and other ports during the month of October totaled 366,121 pounds valued at $339,218, an increase over imports of the previous two months. The imports by ports were as follows: New York, 324,769 pounds with an invoice value of $295,166; Albany, 32,619 pounds with an invoice value of $37,419; Boston, 8,147 pounds with an invoice value of $6,138; Philadelphia, 566 pounds, with an invoice value of $477; and Chicago, 20 pounds, valued at $18. Germany supplied 72 per cent of the October imports and Switzerland 10 per cent, as compared with 62 per cent for Germany for the previous month and 21 per cent for Switzerland.

The five leading dyes by quantity imported during October were:

	Pounds
Rhodamine B Extra (single strength)	19,880
Indanthrene Blue GCD (single strength)	19,832
Brilliant Indigo 4B (single strength)	10,270
Rhodamine 6G Extra (single strength)	10,000
Indanthrene Blue G (single strength)	8,871

POSITION WANTED

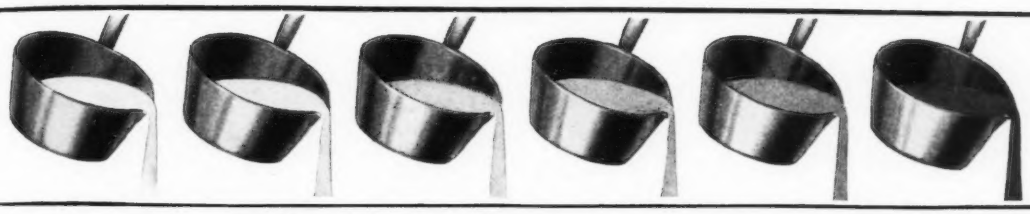
Consulting textile chemist and dyer; experienced in analyzing and testing out dyestuffs, soaps, oils, softeners, gums and chemicals, for their best money value. Also experienced in testing yarns, cloths and silks. Dyeing experience: raw wool and cotton, worsted yarns and cotton, piece dyes of all kinds, union goods, mixed piece dyes made up of cotton, artificial and tram silk, shoddy, rags, stripping and bleaching, carbonizing and mercerizing. American, 45; best of references; familiar with all kinds of dyeing machines. Address Box 316, American Dyestuff Reporter.

DYER WANTED

Man fully familiar with the handling and dyeing of piece goods, especially glove silk jersey cloth. A very good opportunity. Give full details in confidence to Box 319, American Dyestuff Reporter.

TEXTILE CHEMIST

Wanted—Position as textile chemist and dyer; have had experience on hosiery and piece goods; laboratory experience; college training; good references. Reply Box 321, American Dyestuff Reporter.



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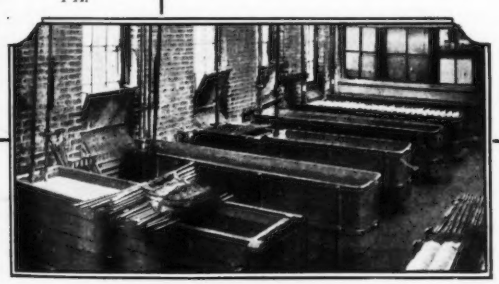
Edwin F. James *

* Mr. Edwin F. James is a recognized authority on the dyeing of Rayon. Your attention is invited to his article "Dyeing of Rayon in the Skein," which appeared in the September 26th issue of Textile World, "Rayon Supplement."

View of Textile Silk Dye Works, Philadelphia, showing Monel Metal equipment made and installed by THE LIBERTY COPPERSMITHING CO. OF PHILADELPHIA, PA.



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